

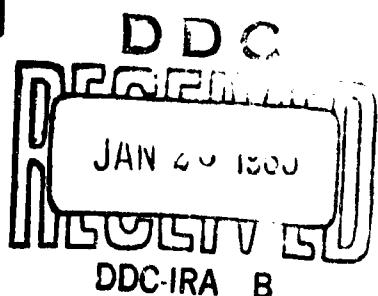
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EFFECT OF JP-5 SULFUR CONTENT ON HOT CORROSION OF SUPER ALLOYS IN MARINE ENVIRONMENT

PROGRESS REPORT NO. 2

NAVY BUWEPS CONTRACT NOw 64-0443-d

139



PHILLIPS PETROLEUM COMPANY

PHILLIPS PETROLEUM COMPANY
RESEARCH DIVISION
BARTLESVILLE, OKLAHOMA

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By

H. T. Quigg
R. M. Schirmer

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Bureau of Naval Weapons Contract N0W 64-0443-d

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S U M M A R Y

An exploratory program was completed to provide a basis for a subsequent factorial experiment to determine whether the maximum sulfur content of 0.40 weight per cent, currently allowed in grade JP-5 aviation turbine fuel, is a safe level for the protection of turbine blade alloys used in high-performance engines.

Specimens of six different superalloys mounted in a cascade holder were exposed to vitiated air from the Phillips 2-inch combustor during a five hour cyclic test. Operating conditions were varied to investigate the effect of gas temperature, pressure and velocity at the test specimens, combustor air-fuel ratio, sea salt ingestion rate, and test specimen position in the cascade. Evaluations were based upon metal loss, scale analysis and metallographic examination of the test specimens.

The desirability of two experiments was shown; one at gas temperatures of 1800 F and above, where fuel sulfur inhibited hot corrosion, and the other below the melting point of sodium sulfate (1623 F), where fuel sulfur accelerated hot corrosion. Analysis of the data obtained indicates that the effects of other test variables on hot corrosion is a function of mass exposure to "sea salt" and fuel sulfur. While test-to-test and stage-to-stage repeatability is good enough in the high temperature range for a factorial experiment based upon triplicate tests, a greater number of tests will be required in the lower temperature range.

A factorial experiment is outlined for three gas temperatures (1800, 2000 and 2200 F), three "sea salt" in air concentrations (0, 1.0 and 10.0 ppm), and three sulfur in fuel concentrations (0, 0.040 and 0.40 weight per cent). Five different superalloys and one coated superalloy will be employed. The extent of corrosive attack will be evaluated from measurements of metal loss and by metallographic examination of the test specimens.

December 31, 1964

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PHILLIPS PETROLEUM COMPANY

BARTLESVILLE, OKLAHOMA

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Navy BuWeps Contract N0w 64-0443-d

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IN MARINE ENVIRONMENT

I. INTRODUCTION

The effect of fuel sulfur on "hot section" durability of aircraft gas turbine engines has been the subject of a number of limited investigations which have been conducted by Phillips Petroleum Company under U. S. Navy Bureau of Naval Weapons Contracts (Ref. 1, 2, 3, 4, 5 and 6). These investigations have been summarized and the problem of hot corrosion in marine environment of superalloys in aircraft engines of advanced design was reviewed in Progress Report No. 3 for Contract N0w 63-0406-d (Ref. 7). This Report (Ref. 7) included results of a statistically designed program used to evaluate the effect of three sulfur concentration levels in the fuel (0.0002, 0.040 and 0.40 weight per cent) at three "sea salt" concentration levels in the air (zero, 1.50 and 15.0 parts per million), and also any sulfur X "sea salt" interactions for two nickel-base alloys (Inconel 713 C and Sierra Metal 200) exposed to vitiated air from the Phillips 2-inch combustor. Test conditions used were

- (1) 56 air-fuel ratio.
- (2) 2000 F gas temperature at the test specimen.
- (3) 15 atmospheres pressure at the test specimen.
- (4) 500 feet per second gas velocity at the test specimen.
- (5) Five hours cyclic operation (55 minutes fuel on and five minutes fuel off).

From this investigation it was concluded that decreasing sulfur concentration in fuel, from the current JP-5 specification maximum of 0.40 to 0.040 weight per cent, did not reduce "sea salt" corrosion significantly. However with the complex interaction found with ingested "sea water", additional data were needed before a recommendation as to the maximum sulfur limit in JP-5 could be made. A test program was proposed to obtain the additional information needed using more superalloys evaluated over a range of gas temperatures.

Exploratory programs have been conducted during the first two periods under Navy BuWeps Contract N0W 64-0443-d to evaluate the effect of operating conditions on hot corrosion. These data will be used to select operating conditions to be used in designing a program to be conducted during the last two quarters which will provide the additional information needed as to the effect of fuel sulfur on hot corrosion of superalloys in a marine environment. During the first quarterly period a cascade test specimen holder was designed and fabricated which permits the evaluation of the effect of sulfur and "sea salt" on hot corrosion under various operating conditions of six superalloys simultaneously. The effect of gas temperature was evaluated using single test specimens of six superalloys at each of six gas temperatures at two levels of fuel sulfur with one level of "sea salt".

During the second quarterly period the exploratory program has included investigations of the effects of:

- (1) Hot gas velocity at the test specimen.
- (2) Hot gas pressure at the test specimen.
- (3) "Sea salt" concentration in the hot gas.
- (4) Air-fuel mixture to combustor.
- (5) Location of test specimen in cascade holder.
- (6) Repeatability of hot corrosion.

II. TEST EQUIPMENT

A. PHILLIPS 2-INCH COMBUSTOR

A schematic diagram of the Phillips 2-inch combustor installation used in this study is shown in Figure 1. Design details of this combustor have been described previously (Ref. 9). Basically, it embodies the principle features of a modern aircraft gas turbine combustor. It was a straight-through, can-type, combustor with fuel atomized by a single, simplex-type nozzle. The flame tube was fabricated from 2-inch, Schedule 40, Inconel pipe, with added internal deflector skirts for film cooling surfaces exposed to the flame.

The supporting test facility has been described previously in detail (Ref. 10). Briefly, air was supplied by rotary Fuller compressors, filtered by a Selas Vape-Sorber, and preheated by a Thermal Research heat exchanger. Fuel was supplied by nitrogen pressurization of its supply tank. Also, "sea water" was supplied by nitrogen pressurization of its supply tank.

The design of the combustor installation provides for easy access to the fuel nozzle, flame tube, and test specimens. The combustor installation was disassembled, inspected, and reconditioned after every test.

The location of the "sea water" injection point in the quench zone of the combustor and the water jacketed exhaust section, shown in Figure 1, were selected on the basis of a previous investigation (Ref. 7).

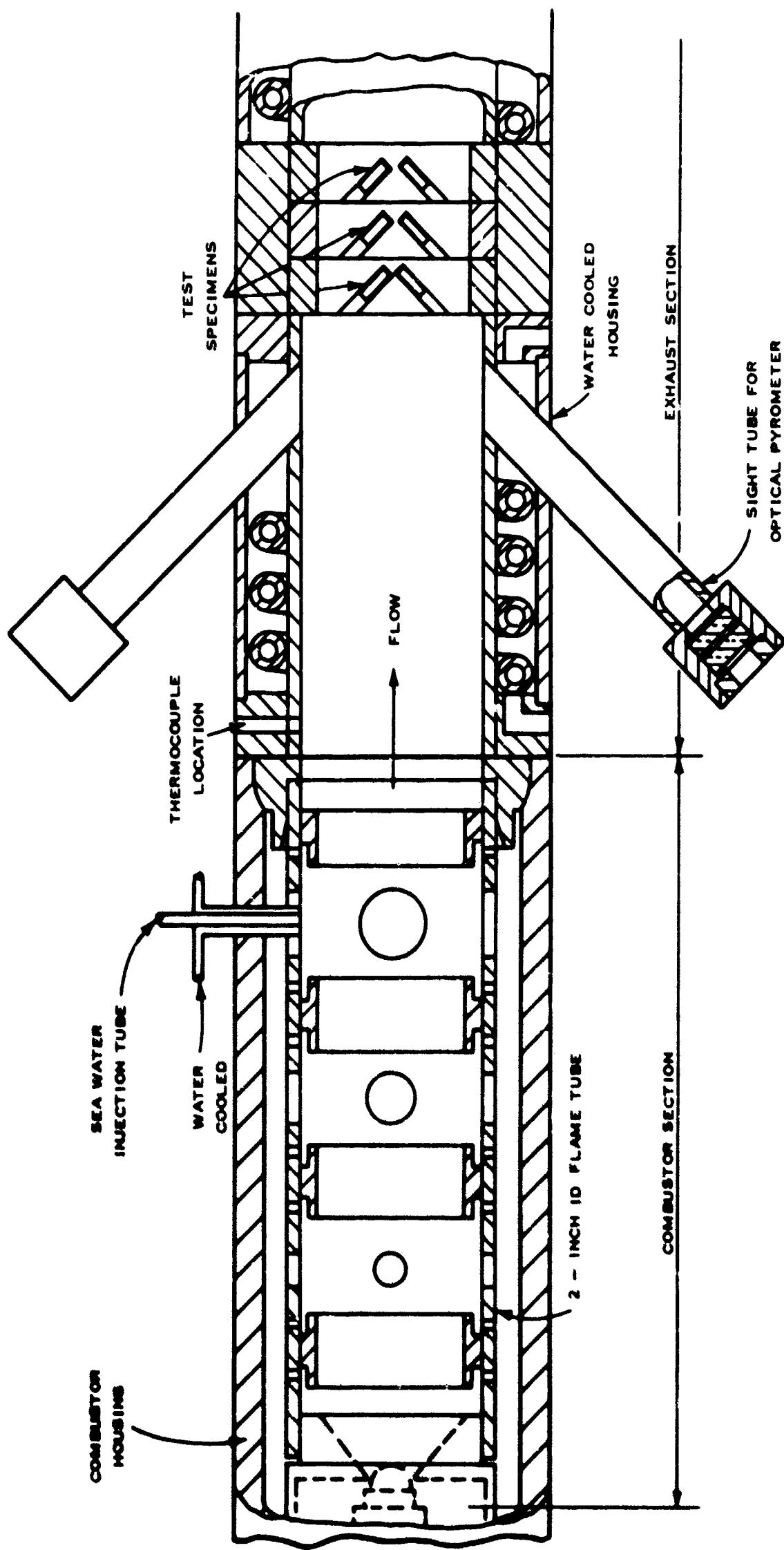


FIGURE 1
PHILLIPS 2 - INCH COMBUSTOR INSTALLATION FOR HOT CORROSION STUDIES

B. SPECIMEN HOLDER

The test specimen holder employed in earlier work (Ref. 7) was modified to permit three holders to be combined in a cascade with each successive holder rotated 120 degrees to prevent channeling of the hot gas flow. The general location with respect to the 2-inch combustor is shown in Figure 1. It is separated from the 2-inch combustor by a six-inch long water cooled spool and is followed by another water cooled spool one foot in length. The holder for a pair of specimens is shown in Figure 2 and the cascade is shown in Figure 3.

The holders maintain the test specimens at an angle of 45 degrees to the axis of the pipe in which they are located. This provides for acceleration of the gas flow over the surface of the test specimens, much as over the turbine blading in an actual engine.

C. SPECIMEN ELECTROCLEANING

Specimen scale or bulk oxide after exposure to hot corrosion was removed by an electrocleaning technique described previously (Ref. 7). Briefly this consists of immersing the specimens in molten sodium hydroxide (750-790 F) and passing about $1/3$ ampere/cm² through the specimens for a period of 10 minutes. This is followed by a water quench.

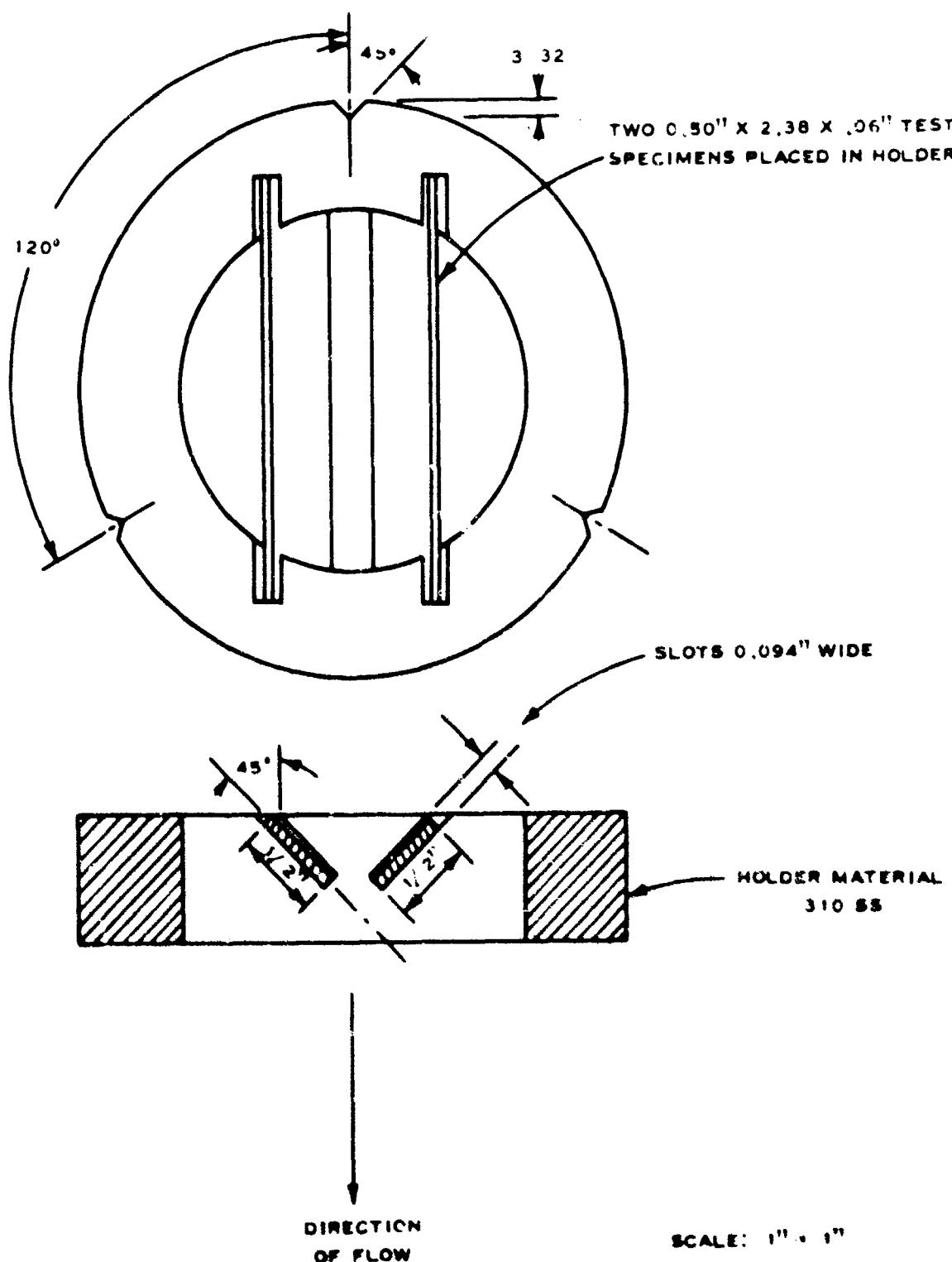


FIGURE 2
TEST SPECIMEN HOLDER FOR PHILLIPS 2 - INCH COMBUSTOR

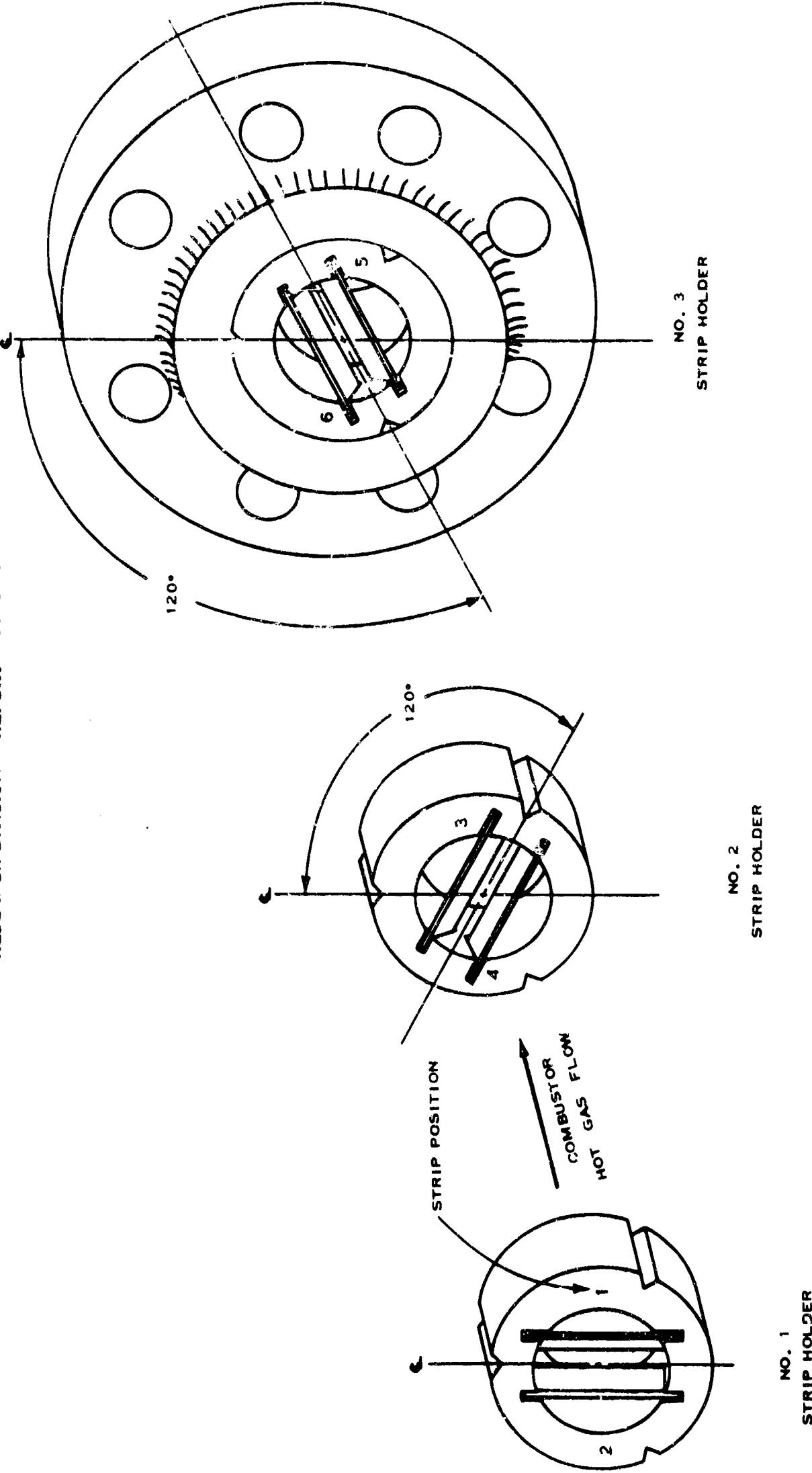


FIGURE 3
CASCADE STRIP HOLDER ASSEMBLY
FOR CORROSION STUDIES

III. TEST MATERIALS

A. TEST FUELS

The base fuel used in this study was a segregated sample of production ASTM Type A aviation turbine fuel. Physical and chemical properties of this fuel are shown in Table I.

The base fuel was essentially sulfur-free, containing only 0.0002 weight per cent sulfur. The higher sulfur content test fuel was produced by blending to 0.40 weight per cent sulfur using ditertiary butyl disulfide.

B. "SEA WATER" (a)

A synthetic "sea water" was used in this study. Its formulation was taken from the Standard Method of Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water, ASTM Designation D-665-60. The components and their concentrations are shown in Table II.

C. SUPERALLOYS

Eight different superalloys were used as test specimens during these studies. In the study of the effect of temperature during the first quarterly period four nickel-base (Udimet 700, Hastelloy R-235, Sierra Metal 200, and Udimet 500) and two cobalt-base (Stellite 31 and Haynes Alloy 25) superalloys were used. For the remainder of the investigation conducted during the second quarterly period two nickel-based (Rene' 41 and Waspalloy) superalloys were substituted for Stellite 31 and Haynes Alloy 25 because of availability. Three of the superalloys were prepared as investment castings (Udimet 700, Stellite 31, and Sierra Metal 200) while the other five were cut from sheets. These metals were selected to represent a wide range of chromium contents. The chemical analyses for the superalloys used are shown in Table III.

(a) "Sea water" and "sea salt" (i.e. in quotes) are used throughout this report to indicate the synthetic composition shown in Table II.

TABLE I

PHYSICAL AND CHEMICAL PROPERTIES OF TEST FUELS

<u>Distillation Temperature, F</u>	<u>Test Fuel Base (a)</u>
Initial Boiling Point	329
5 Volume per cent evaporated	344
10	350
20	359
30	368
40	377
50	388
60	400
70	417
80	435
90	460
95	478
End Point	498
Gravity, degrees API	46.2
Gum, milligrams per 100 milliliters	0.2
Smoke Point, millimeters	26.2
Composition, weight per cent	
Sulfur	0.0002 (b)
Metals (c)	
Iron	< 0.0001
Vanadium	< 0.0001
Nickel	< 0.0001
Copper	< 0.0001
Hydrocarbon Types	
Normal Paraffins	27 (d)
Isoparaffins	23 (d)
Cycloparaffins	36 (d)
Olefins	0 (d)
Aromatics	14

- (a) Segregated sample (BJ63-8-G49) of production ASTM Type A Aviation turbine fuel, processed from West Texas crude and finished by hydrotreating.
- (b) Higher sulfur content test fuel obtained by blending to desired level using ditertiary butyl disulfide.
- (c) X-ray fluorescence analysis.
- (d) Typical value for this product.

TABLE II
COMPOSITION OF ASTM D665 SYNTHETIC "SEA WATER"

<u>Salt (a)</u>	<u>Formula</u>	<u>grams per liter (b)</u>
Sodium Chloride	NaCl	24.54
Magnesium Chloride	MgCl ₂ .6H ₂ O	11.10
Sodium Sulfate	Na ₂ SO ₄	4.09
Calcium Chloride	CaCl ₂	1.16
Potassium Chloride	KCl	0.69
Sodium Bicarbonate	NaHCO ₃	0.20
Potassium Bromide	KBr	0.10
Boric Acid	H ₃ BO ₃	0.03
Strontium Chloride	SrCl ₂ .6H ₂ O	0.04
Sodium Fluoride	NaF	0.003
	TOTAL	41.953

(a) Use cp chemicals.

(b) Use distilled water.

TABLE III
 COMPOSITION OF SUPERALLOY TEST SPECIMENS

Allotting Elements	Chemical Analysis, Per Cent							
	Udinet 700 (a)	Stellite 31 (a)	Hastelloy R-235 (b)	25 (b)(c)	Udinet 200 (a)	Udinet 500 (b)	Rene' 41(b)	Waspalloy(b)
Nickel	Balance (53.05)	10.41	Balance (63.91)	10.0	Balance (60.34)	Balance (51.04)	Balance (54.37)	Balance (57.44)
Cobalt	18.54	Balance (53.62)	0.38	Balance (49.4)	9.80	18.7	10.69	13.2
Chromium	14.89	25.05	15.29	20.0	9.14	19.0	18.33	19.5
Molybdenum	5.10	--	5.48	--	--	4.35	9.69	4.41
Tungsten	--	7.68	--	15.0	12.12	--	--	--
Aluminum	4.32	--	2.05	--	4.78	3.10	1.54	1.23
Titanium	3.02	--	2.48	--	2.00	2.99	3.15	3.10
Manganese	Nil	0.73	0.03	1.5	0.04	< 0.10	0.05	0.01
Iron	0.75	1.37	9.96	3.0	0.53	0.36	1.90	0.95
Zirconium	0.042	--	--	--	0.068	< 0.01	--	--
Columbium	--	--	--	--	0.99	--	--	--
Silicon	0.18	0.62	0.26	1.0	0.01	0.15	0.16	0.3
Boron	0.031	--	--	--	0.015	0.003	0.005	0.002
Sulfur	0.006	0.012	0.009	--	--	0.005	0.009	0.003
Carbon	0.06	0.49	0.15	0.1	0.17	0.09	0.10	0.053
Phosphorus	0.001	0.015	0.001	--	--	--	--	0.003
Copper	0.02	--	--	--	< 0.10	--	--	0.01

(a) Investment casting. (b) Cut from sheet material. (c) Typical analysis.

IV. TEST PROGRAM

In most cases in these exploratory programs single test specimens of each of six superalloys have been mounted in a cascade for exposure to hot gases at the test conditions being investigated. As shown in Figure 3 the cascade consists of three stages each holding two test specimens. It may be observed that only the specimens in the first stage are exposed to hot gases only, while those in the second and third stages may also be exposed to corrosion products from the preceding superalloys. The position of a specific superalloy within the cascade is held constant throughout the programs.

The test procedure consists of a five-hour cyclic test with 55 minutes of exposure of the test specimens to hot gases followed by 5 minutes with fuel turned off. On completion of the test, samples of deposits are removed by scraping and saved for analyses. The test specimens are then electro-chemically cleaned using a procedure previously described (Ref. 7) and loss of weight determined.

Preliminary data on the effect of temperature on hot corrosion were presented in Progress Report No. 1 (Ref. 8) and are included in this report to summarize the exploratory study of operating variables. The effect of temperature was studied over the range of 1200 to 2200 F with 0.0002 and 0.40 weight per cent fuel sulfur and 10 ppm "sea salt" in combustor air. Combustor pressure was maintained at 15 atmospheres with a mass air flow of 5400 pounds per hour. Combustor inlet air temperature and fuel flow were adjusted to give the desired gas temperature. Since a reversal in the effect of fuel sulfur content on hot corrosion in a marine environment was indicated with increasing temperature, two temperature-fuel sulfur combinations (1500°F with 0.40 and 2000°F with 0.0002 per cent) were selected to maximize hot corrosion in the study of other test variables.

The operating variables studied include combustor pressure (5 and 15 atmospheres), gas velocity at the test specimen (50 to 640 feet per second), "sea salt" in combustor air (2.0 and 10.0 ppm) and air fuel ratio (50 and 75). The effect of position of the test specimens in the cascade on metal loss has also been investigated.

A summary of the operating variables investigated during this test program and test numbers is shown in Table IV and detailed operating conditions for these tests are shown in Appendix I. Where possible one test has been used for multiple comparisons of operating variables.

TABLE IV
SUMMARY OF OPERATING VARIABLES STUDIED

<u>Variables Studied</u>	<u>Test Numbers</u>
<u>Temperature, °F</u> 1200, 1400, 1600, 1800, 2000, 2200 (a) 0.0002 per cent sulfur (b) 0.40 per cent sulfur	8, 9, 14, 15, 18, 19 7, 10, 13, 16, 17, 20
<u>Combustor Pressure, Atmospheres</u> 5, 15	25, 26, 27, 28
<u>Gas Velocity, feet per second</u> 50, 250, 500, 644	23, 24, 27, 28, 31, 32, 33
<u>Air Fuel Ratio</u> 50, 75	34, 38
<u>"Sea Salt" Ingestion Rate, ppm</u> 2.0, 10.0	27, 28, 34, 35, 36, 37, 38, 39, 41
<u>Cascade Specimen Holder</u> Position of specimens in cascade and repeatability of metal loss	34, 35, 36, 37, 38, 39, 41

V. DISCUSSION OF RESULTS

A. Metal Weight Loss.

1. Temperature.

The metal loss data obtained in an exploratory program on the effect of temperature are shown in Table V expressed as weight loss per unit area and in Table VI expressed as per cent weight loss of the test specimen. Metal loss as a function of temperature for two fuel-sulfur concentrations are shown in Figures 4 through 9. An examination of these data show that with each of the superalloys, metal loss was inhibited by the fuel sulfur content at temperatures of 1800 F and above. At 1600 F and below the data are not consistent for all of the superalloys; however, with Hastelloy R-235 and to a lesser degree with Sierra Metal 200, Udimet 700 and Udimet 500 sulfur in the fuel increases metal loss. This reversal of sulfur effect may be associated with the presence of solid sodium sulfate on the test specimens. The freezing point of sodium sulfate is 1623 F. Although air-fuel ratio varied from 150 at 1200 F to 50 at 2200 which resulted in an increase in sulfur to "sea salt" ratio, comparisons of low (0.0002 weight per cent) and high (0.40 weight per cent) sulfur fuels at a given temperature would not be affected.

2. Combustor Pressure.

The metal weight loss data for single tests of six superalloys at 5 and 15 atmospheres pressure for two operating conditions are shown in Table VII expressed as weight loss per unit area and as per cent weight loss of the test specimen. Metal losses as a function of combustor pressure for 1500 F exhaust gas temperature and 0.40 per cent fuel sulfur and for 2000 F exhaust gas temperature with 0.0002 per cent fuel sulfur are summarized in Figures 10 and 11. Metal loss is more severe at the high temperature-low sulfur conditions than at the low temperature-high sulfur conditions with one exception at 15 atmospheres combustor pressure. While metal loss varies for the superalloys an increase in combustor pressure accelerates hot corrosion for those alloys subject to attack.

TABLE V
WEIGHT LOSS (lb/cm²) OF SUPERALLOY TEST SPECIMENS

Run No.	Gas Temp., F.	Fuel Sulfur Wt. %	Weight Loss, lb per cm ² (a)		Sierra Metal 200	Udimet 500		
			Hastelloy R-235					
			200	31				
19	1200	0.0002	8.7	3.0	11.5	7.4		
20	1200	0.40	42.4	3.8	116.7	3.5		
18	1400	0.0002	36.4	5.2	72.8	11.2		
17	1400	0.40	6.8	3.4	174.4	5.8		
14	1600	0.0002	58.5	11.4	169.2	10.0		
13	1600	0.40	1.7	5.8	343.6 (b)	11.5		
15	1800	0.0002	84.3	16.3	112.0	10.8		
16	1800	0.40	8.4	2.7	9.1	8.7		
8	2000	0.0002	100.1	30.4	72.9	34.8		
7	2000	0.40	5.6	11.6	9.5	11.9		
9	2200	0.0002	107.2	82.1	147.9	234.6		
10	2200	0.40	24.1(d)	24.5	66.8	31.0		

(a) Area of specimen = 17.67 cm².

(b) Part of specimen burned out.

(c) Center missing.

(d) Specimen broken.

(e) Specimen missing at end of test.

TABLE VI
PER CENT METAL WEIGHT LOSS OF SUPERALLOY TEST SPECIMENS

Run No.	Gas Temp., F.	Fuel Sulfur, Wt. %	Udinet 700	Stellite 31	Per Cent Metal Weight Loss			Udinet 500
					Hastelloy R-235	Haynes Alloy 25	Sierra Metal 200	
19	1200	0.0002	1.7	0.5	1.9	1.2	0.7	2.0
20	1200	0.40	7.7	0.6	19.6	0.6	2.0	2.2
18	1400	0.0002	7.0	0.9	12.3	1.8	3.0	2.1
17	1400	0.40	1.2	0.6	29.4	1.0	11.0	10.2
14	1600	0.0002	10.8	1.8	28.6(1)	1.6	16.4	6.6
13	1600	0.40	0.3	0.9	57.5(1)	1.9	11.4	1.5
15	1800	0.0002	16.2	2.8	19.0	1.8	36.3	1.7
16	1800	0.40	1.6	0.4	1.5	1.5	13.3	0.7
8	2000	0.0002	18.8	5.2	12.2	5.8	79.8(2)	2.9
7	2000	0.40	1.0	2.0	1.6	2.0	1.3	1.4
9	2200	0.0002	19.5	14.0	25.0	40.5	88.2	13.1
10	2200	0.40	4.4(3)	4.0	11.4	5.2	16.3	—(4)

- (1) Part of specimen burned out.
- (2) Center missing.
- (3) Specimen broken.
- (4) Specimen missing at end of test.

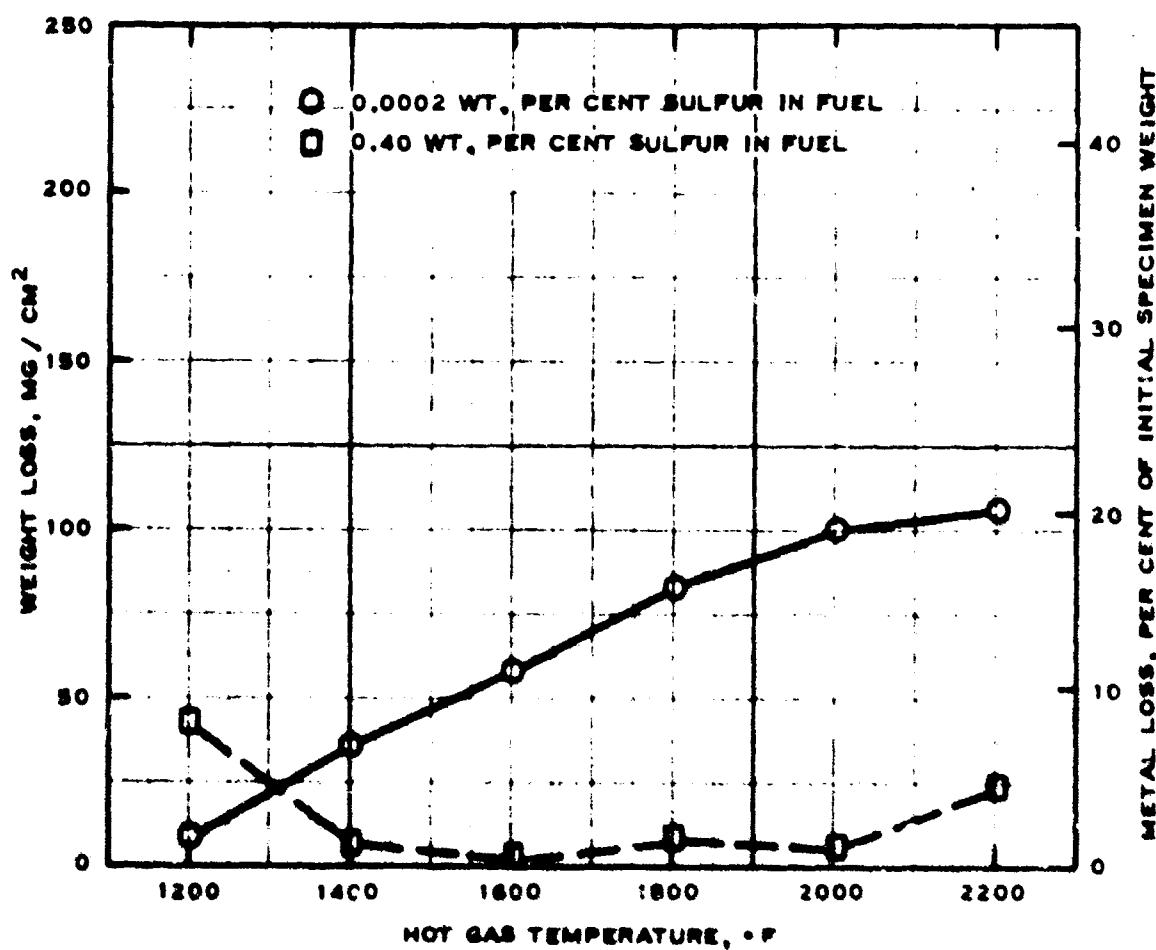


FIGURE 4
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF UDIMET 700 IN MARINE ENVIRONMENT

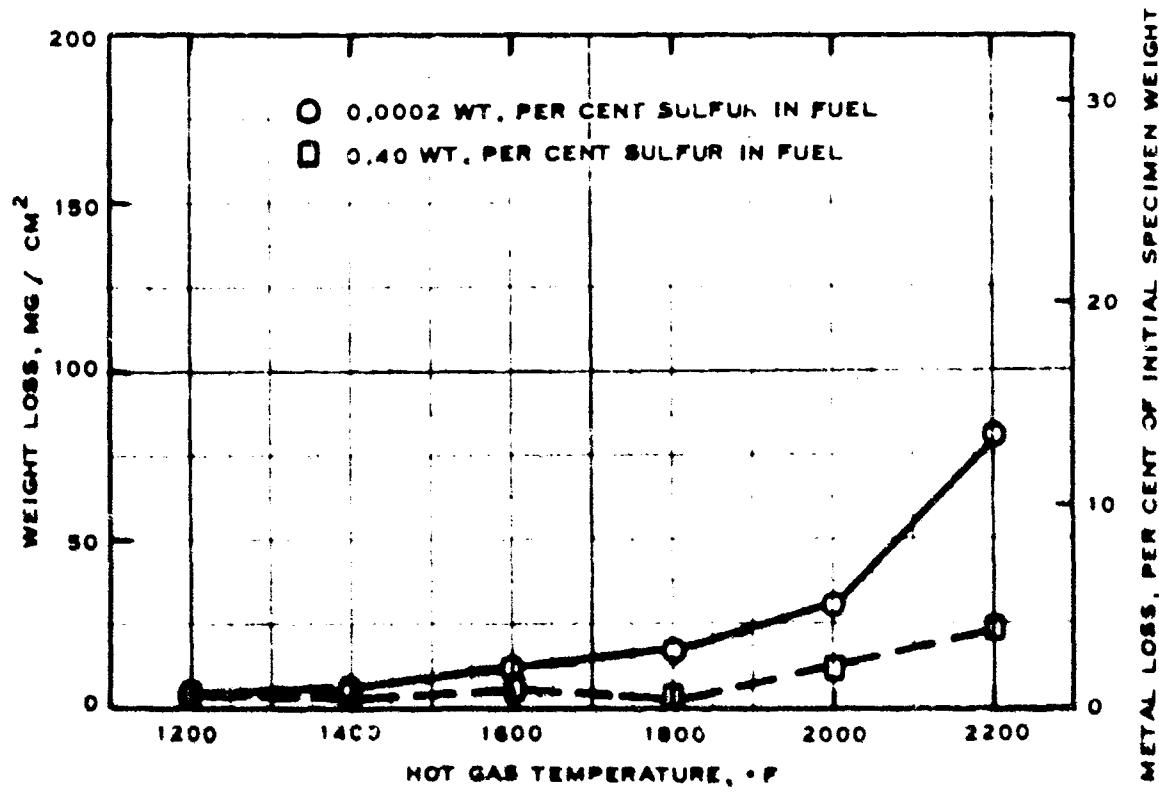


FIGURE 5
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF STELLITE 31 IN MARINE ENVIRONMENT

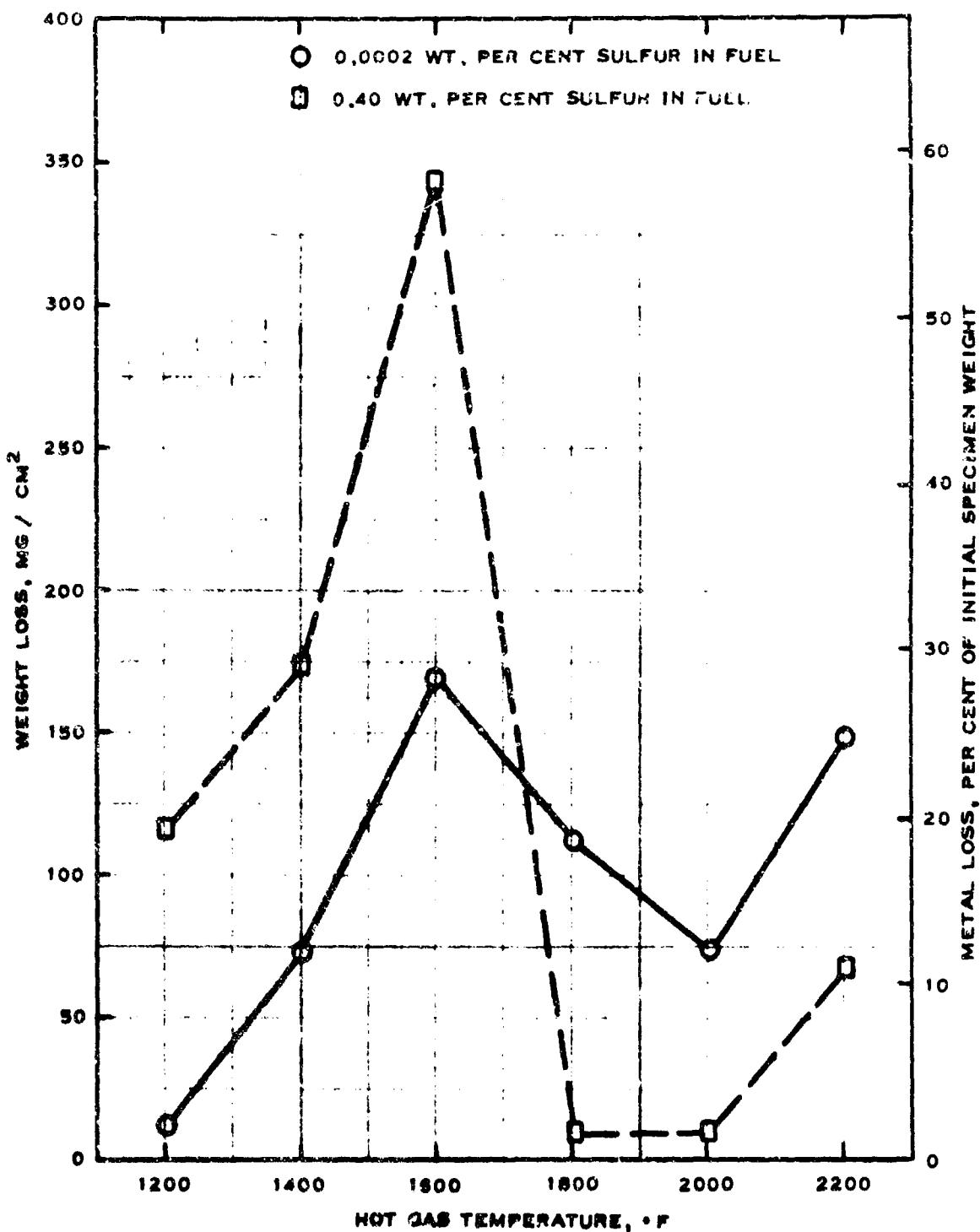


FIGURE 6
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS CORROSION
OF HASTELLOY R - 235 IN MARINE ENVIRONMENT

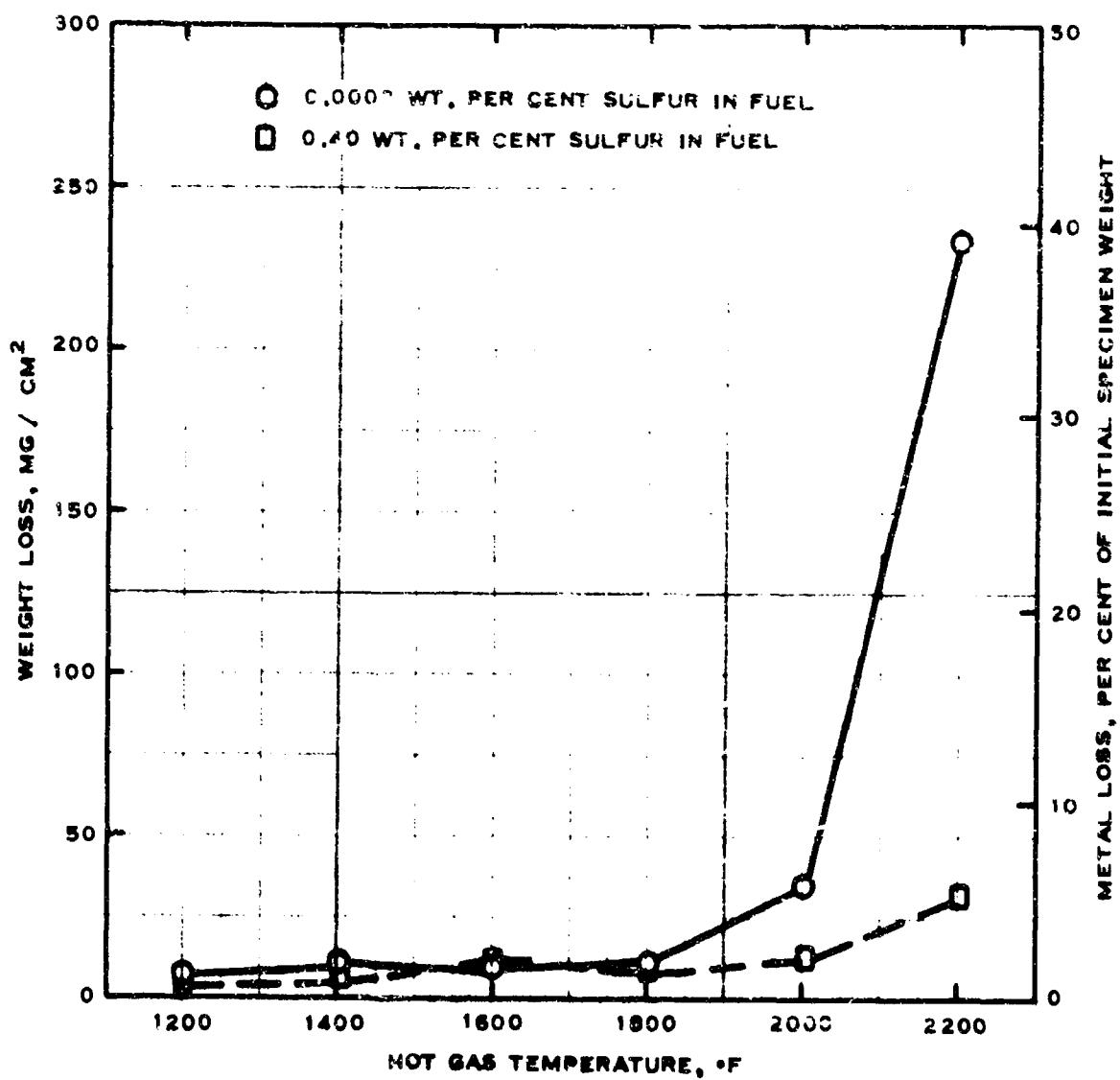


FIGURE 7
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF HAYNES ALLOY 25 IN MARINE ENVIRONMENT

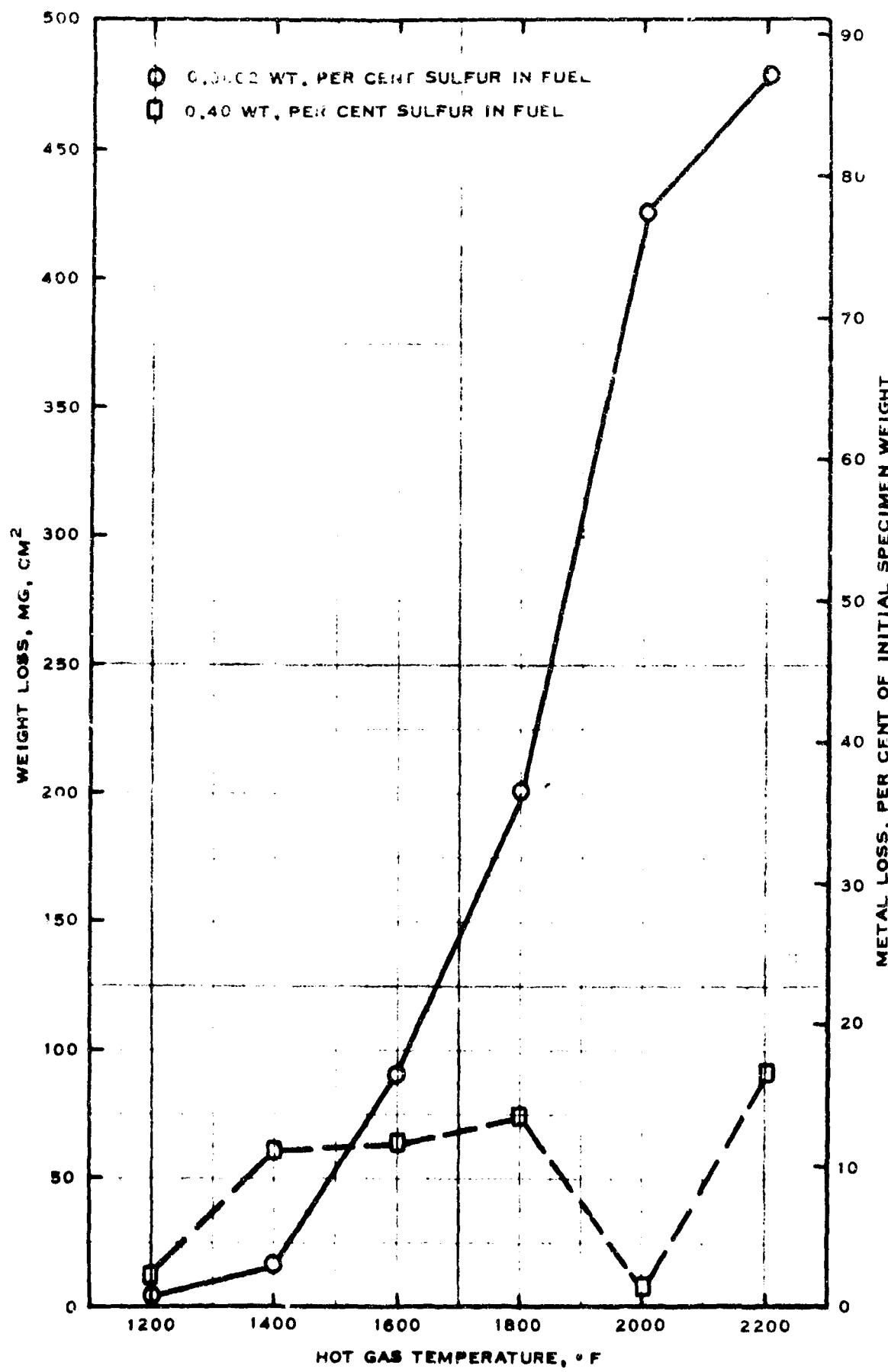


FIGURE 8
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF SIERRA METAL 200 IN MARINE ENVIRONMENT

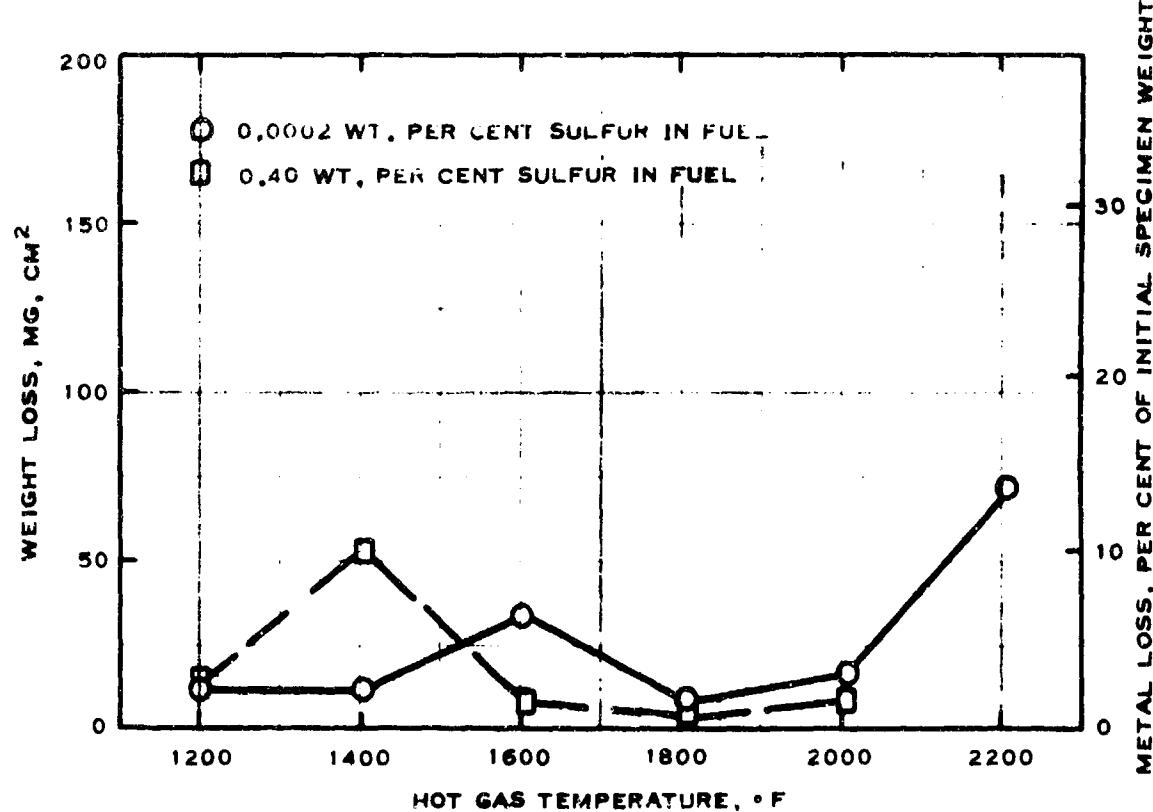


FIGURE 9
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF UDIMET 500 IN MARINE ENVIRONMENT

TABLE VII

EFFECT OF COMBUSTOR PRESSURE ON METAL LOSS OF SUPERALLOY TEST SPECIMENS

<u>Superalloy</u>	<u>Test Conditions</u>	<u>Weight Loss, Mg per cm²</u>		<u>Per Cent Weight Loss</u>	
		<u>5 Atm.</u>	<u>15 Atm.</u>	<u>5 Atm.</u>	<u>15 Atm.</u>
Udimet 700	(a)	0.2	1.2	0.0	0.2
Rene' 41	(a)	0.6	0.8	0.1	0.1
Waspalloy	(a)	1.7	6.0	0.3	1.0
Hastelloy R-235	(a)	12.4	259.0	2.1	43.8
Sierra Metal 200	(a)	3.6	125.6	0.7	22.6
Udimet 500	(a)	2.6	—(c)	0.5	—(c)
Udimet 700	(b)	41.8	112.6	7.6	20.8
Rene' 41	(b)	21.2	8.7	3.4	1.4
Waspalloy	(b)	14.7	8.0	2.4	1.3
Hastelloy R-235	(b)	23.9	52.4	4.0	8.8
Sierra Metal 200	(b)	143.5	252.1	25.0	45.4
Udimet 500	(b)	17.7	7.6	3.5	1.5

(a) Test No. 25 & 27 Gas Temp. 1500 F, Fuel Sulfur 0.40 wt. %, "sea salt" in combustor air 10 ppm; Gas Velocity at Test Specimen 500 FPS.

(b) Test No. 26 & 28 Gas Temp. 2000 F, Fuel Sulfur 0.0002 wt. %, "sea salt" in combustor air 10 ppm, Gas Velocity at Test Specimen 500 FPS.

(c) Specimen missing at end of test.

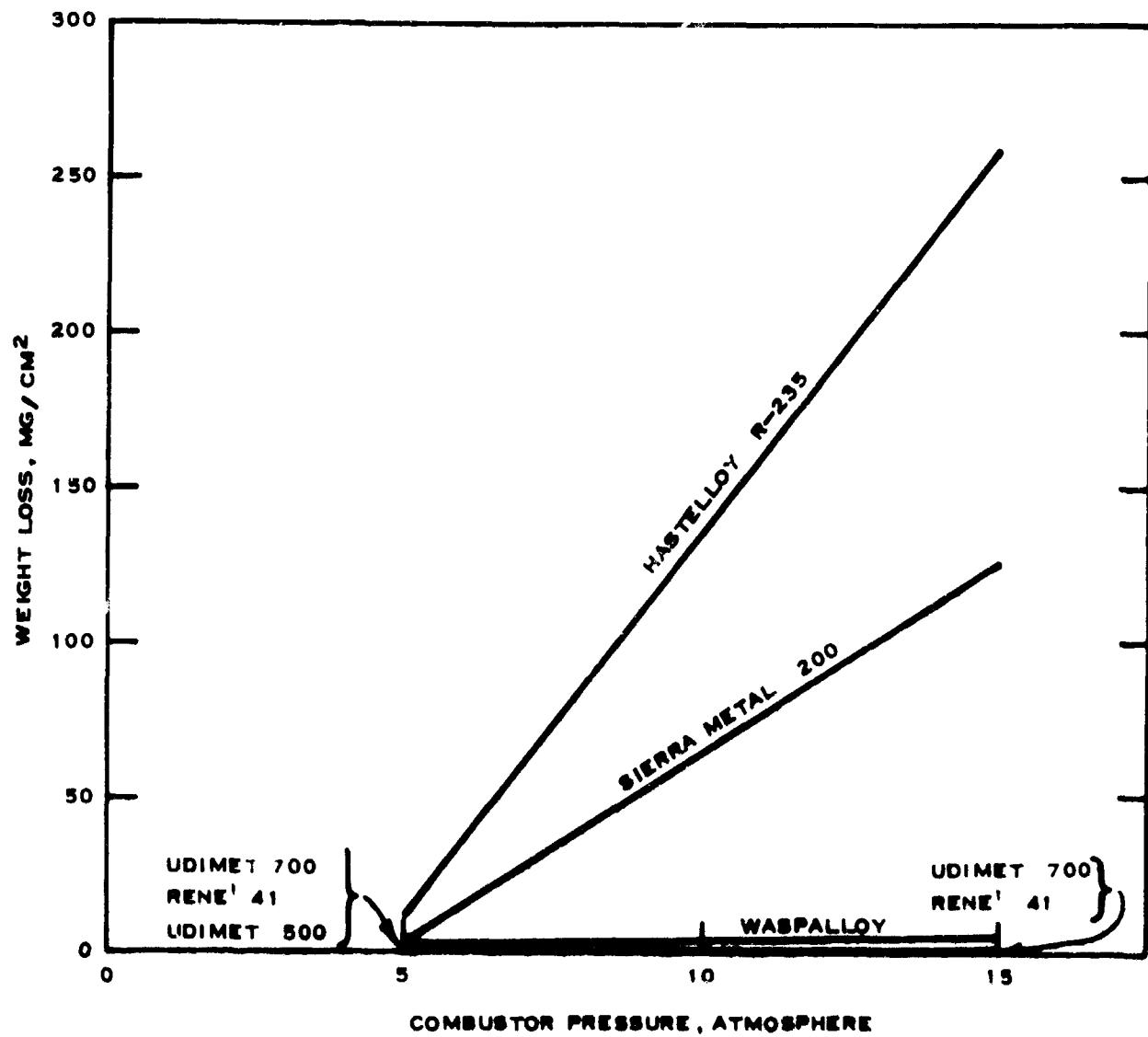


FIGURE 10
EFFECT OF COMBUSTOR PRESSURE ON CORROSION OF SUPERALLOYS
AT 1500 F GAS TEMPERATURE WITH 0.40 WT PER CENT FUEL
SULFUR IN MARINE ENVIRONMENT

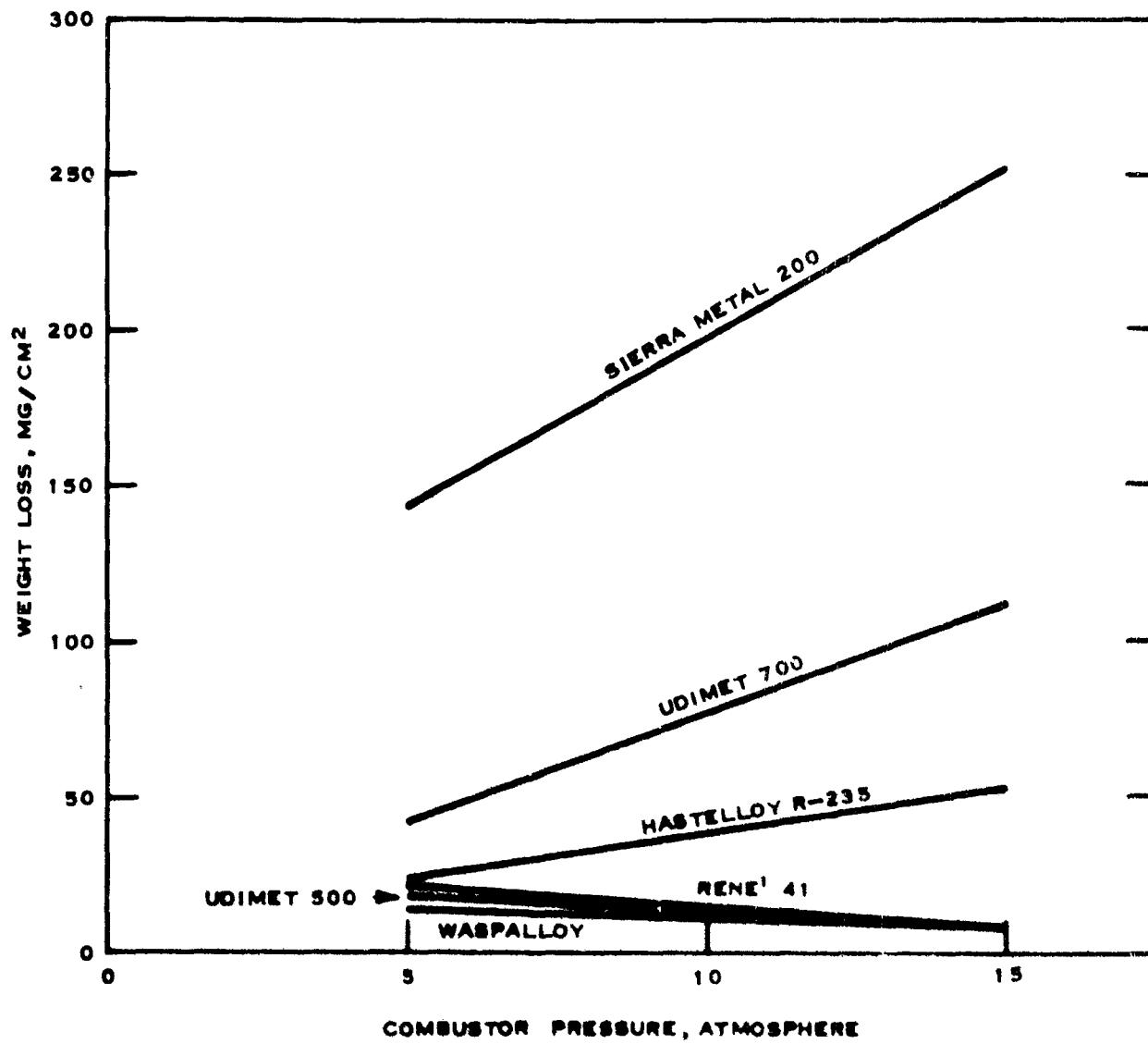


FIGURE 11
EFFECT OF COMBUSTOR PRESSURE ON CORROSION OF SUPERALLOYS
AT 2000 F GAS TEMPERATURE WITH 0.0002 WT PER CENT FUEL
SULFUR IN MARINE ENVIRONMENT

3. Gas Velocity.

The effect of gas velocity on metal loss per unit area is shown in Table VIII and on metal loss as a per cent of specimen weight in Table IX. Data for six superalloys at two exhaust gas temperature-fuel sulfur conditions are summarized in Figures 12 and 13. In most cases the high temperature-low sulfur conditions are more severe than the low temperature-high sulfur conditions. For those superalloys subject to hot corrosion increasing gas velocity increases metal loss.

4. Air-Fuel Ratio.

The effect of air-fuel ratio on metal loss of Udimet 700 at 2000 F gas temperature with 0.0002 weight per cent fuel sulfur and 10.0 ppm "sea salt" in combustor air is shown in Table X. Changing air-fuel ratio from 50 to 75 does not effect metal loss of Udimet 700.

5. "Sea Salt" Ingestion Rate.

Table XI shows the effect of "sea salt" ingestion rate on hot corrosion of Hastelloy R-235 at 1500 F with 0.40 per cent fuel sulfur and of Hastelloy R-235 and Udimet 700 at 2000 F with 0.0002 per cent fuel sulfur. Directionally an increase in "sea salt" ingestion rate from 2.0 to 10.0 ppm in combustor air increases hot corrosion; however, the magnitude of the increase varies with metals, temperature and fuel sulfur.

6. Cascade Position and Repeatability.

In designing a test program information is needed on test repeatability since this will indicate the number of tests needed to permit detection of significant effects. The effect of cascade position on hot corrosion of a test specimen will also influence the number of tests required in a program. To obtain data on the effect of cascade position and repeatability of metal loss, tests have been conducted using multiple test specimens of Hastelloy R-235 or Udimet 700 in the cascade. Metal loss data for these tests are shown in Table XIII expressed as weight loss per unit area and as per cent weight of the test specimen.

TABLE VIII

EFFECT OF GAS VELOCITY ON METAL LOSS (MG PER CM²) OF SUPERALLOY TEST SPECIMENS

Superalloy	Test Conditions	Weight Loss, Mg per cm ²			
		50 FPS	250 FPS	500 FPS	644 FPS
Udimet 700	(a)	6.3	1.1	1.2	---
Rene' 41	(a)	3.8	0.5	0.8	---
Waspalloy	(a)	1.2	0.6	6.0	---
Hastelloy R-235	(a)	1.2	49.3	259.0	---
Sierra Metal 200	(a)	8.0	12.9	125.6	---
Udimet 500	(a)	5.3	7.6	--(c)	---
Udimet 700	(b)	3.3	50.2	112.6	64.4
Rene' 41	(b)	6.7	40.5	8.7	36.4
Waspalloy	(b)	3.2	23.9	8.0	74.7
Hastelloy R-235	(b)	2.1	36.4	52.4	47.2
Sierra Metal 200	(b)	15.1	355.3	252.1	--(c)
Udimet 500	(b)	3.6	19.4	7.6	45.7

(a) Test No. 23, 33 & 27 Gas Temp. 1500 F, Fuel Sulfur 0.40 wt. %, "sea salt" in combustor air 10 ppm. Combustor Pressure 15 Atm.

(b) Test No. 24, 32, 28 & 31 Gas Temp. 2000 F, Fuel Sulfur 0.0002 wt. %, "sea salt" in combustor air 10 ppm, Combustor Pressure 15 Atm.

(c) Specimen missing at end of test.

TABLE IX

EFFECT OF GAS VELOCITY ON METAL LOSS (PER CENT WEIGHT LOSS) OF SUPERALLOY

TEST SPECIMENS

<u>Superalloy</u>	<u>Test Conditions</u>	<u>Per Cent Weight Loss</u>			
		<u>50 FPS</u>	<u>250 FPS</u>	<u>500 FPS</u>	<u>644 FPS</u>
Udimet 700	(a)	1.2	0.2	0.2	---
Rene' 41	(a)	0.6	0.1	0.1	--
Waspalloy	(a)	0.2	0.1	1.0	--
Hastelloy R-235	(a)	0.2	8.6	43.8	--
Sierra Metal 200	(a)	1.4	2.3	22.6	--
Udimet 500	(a)	1.0	1.5	--(c)	--
Udimet 700	(b)	0.6	9.3	20.8	12.1
Rene' 41	(b)	1.1	6.4	1.4	5.8
Waspalloy	(b)	0.5	3.9	1.3	11.8
Hastelloy R-235	(b)	0.4	6.1	8.8	7.9
Sierra Metal 200	(b)	2.7	65.9	45.4	--(c)
Udimet 500	(b)	0.7	3.8	1.5	9.1

(a) Test No. 23, 33 & 27 Gas Temp. 1500 F, Fuel Sulfur 0.40 wt. %, "sea salt" in combustor air 10 ppm, Combustor Pressure 15 Atm.

(b) Test No. 24, 32, 28 & 31 Gas Temp. 2000 F, Fuel Sulfur 0.0002 wt. %, "sea salt" in combustor air 10 ppm, Combustor Pressure 15 Atm.

(c) Specimen missing at end of test.

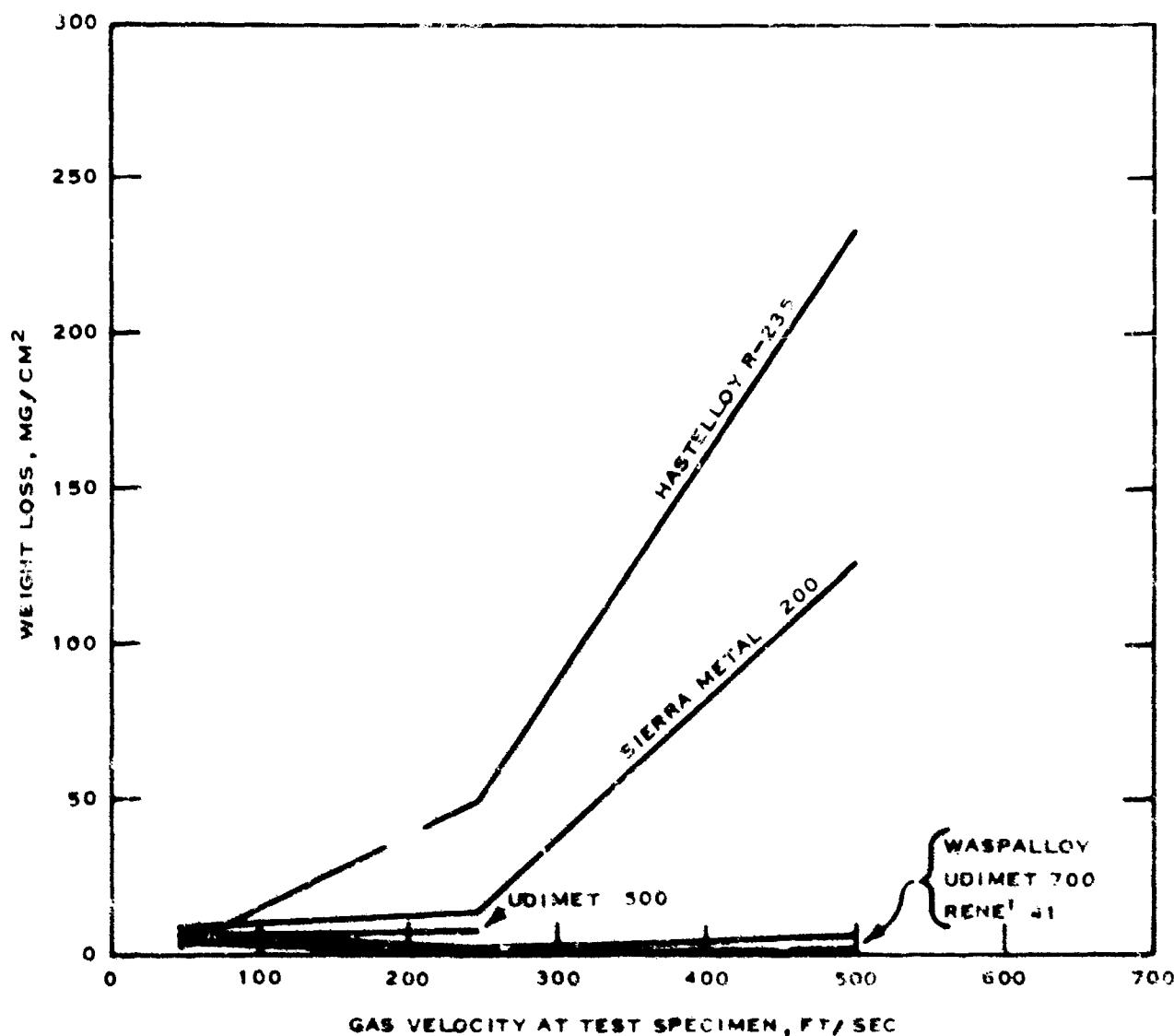


FIGURE 12
EFFECT OF GAS VELOCITY ON CORROSION OF SUPERALLOYS AT
1500 F GAS TEMPERATURE WITH 0.40 WT. PERCENT FUEL SULFUR
IN MARINE ENVIRONMENT

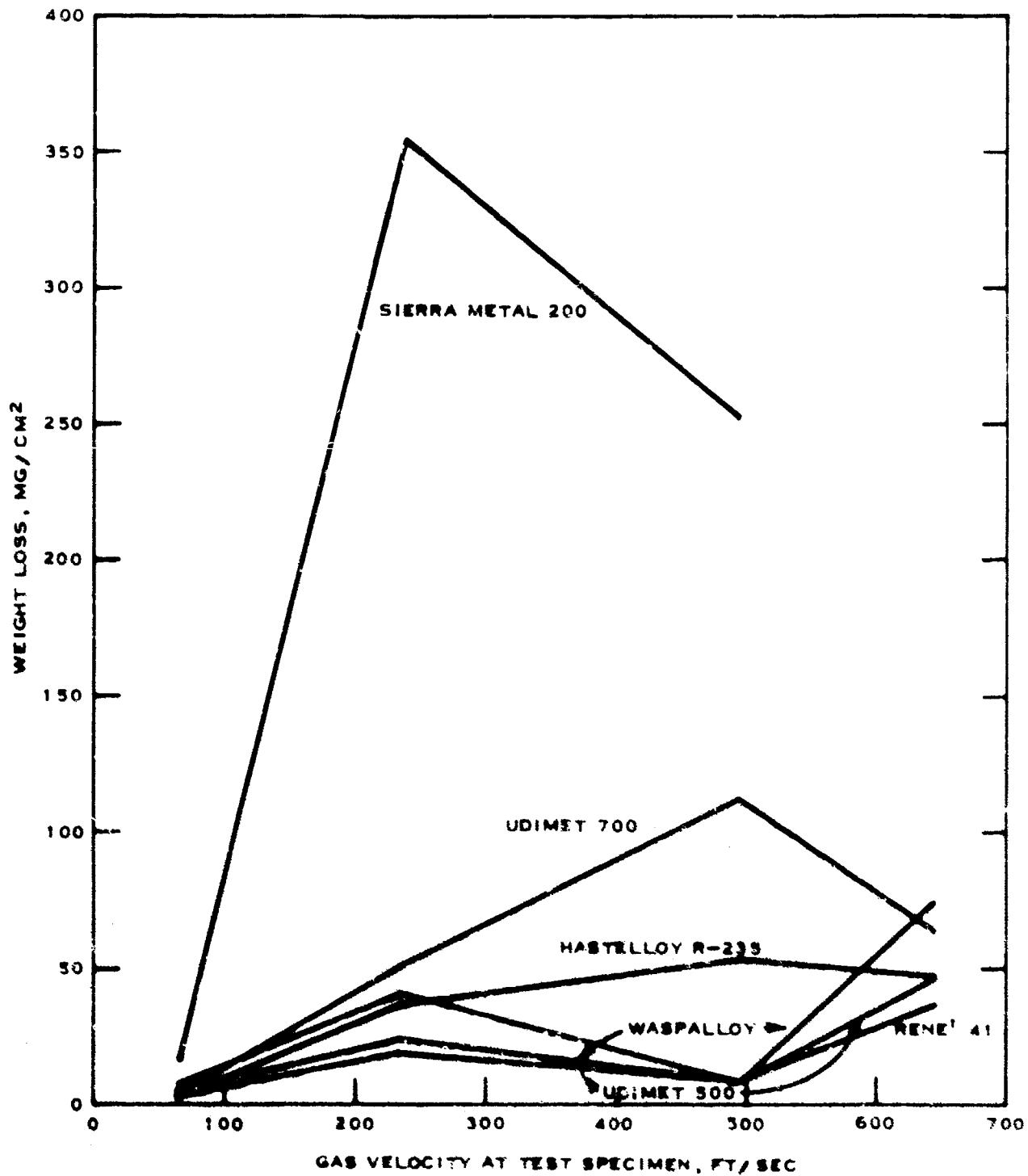


FIGURE 13
EFFECT OF GAS VELOCITY ON CORROSION OF SUPERALLOYS AT
2000 F GAS TEMPERATURE WITH 0.0002 WT PER CENT FUEL SULFUR
IN MARINE ENVIRONMENT

TABLE X
EFFECT OF AIR FUEL RATIO ON METAL LOSS OF UDIMENT 700

Run No.	Air Fuel (a) Ratio	Weight Loss, Mg. per cm ²						Per Cent Metal Weight Loss
		Stage 1 1	Stage 2 2	Stage 3 3	Stage 4 4	Stage 5 5	Stage 6 6	
34	50	110.3	115.4	92.8	129.3	97.5	80.4	20.3
38 (b)	75	87.7	--	86.2	--	--(c)	--	16.1

(a) Gas Temp. 2000 F, Fuel Sulfur 0.0002 wt. %, "sea salt" in combustor air 10 ppm, Gas Velocity at test specimen 500 FPS, Combustor Pressure 15 Atm.

(b) Test duration 4½ rather than 5 hours.

(c) Specimen missing at end of test.

TABLE XI
EFFECT OF "SEA SALT" INGESTION RATE ON METAL WEIGHT LOSS OF SUPERALLOY
TEST SPECIMENS

Run No.	Gas Temp., F.	Fuel Sulfur, Wt. %	"Sea Salt", ppm	Weight Loss, Mg. per cm ²						Per Cent Metal Weight Loss							
				Stage 1			Stage 2			Stage 3			Stage 1			Stage 2	
				1	2	3	4	5	6	1	2	3	4	5	6	5	6
35	1500	0.40	10	57.0	62.5	116.0	Hastelloy	R-235	57.2	9.9	10.8	20.2	24.2	23.5	9.9	—	—
39	1500	0.40	10	44.6	46.8	112.0	389.9	225.1	141.2	7.8	8.2	19.6	68.1	38.8	25.4	—	—
41	1500	0.40	10	14.7	96.4	282.9	200.0	388.2	103.2	2.6	16.8	49.2	34.7	67.3	18.0	—	—
27	1500	0.40	10	—	—	259.0	—	—	—	—	—	—	43.8	—	—	—	—
37	1500	0.40	2	0.7	0.8	0.3	0.9	0.8	0.8	0.1	0.2	0.0	0.2	0.2	0.2	0.2	0.2
28	2000	0.0002	10	—	—	59.7	—	52.4	—	—	—	—	—	8.8	—	—	—
38	2000	0.0002	10	—	—	59.7	—	57.7	—	37.7	—	10.5	—	10.2	—	—	6.6
36	2000	0.0002	2	—	32.7	—	44.5	—	23.5	—	5.7	—	7.8	—	7.8	—	4.1
										Udinet 700							
28	2000	0.0002	10	112.6	—	—	—	—	—	20.8	—	—	—	—	—	—	—
38	2000	0.0002	10	87.7	—	86.2	—	—	—	16.1	—	16.0	—	—	—	—	—
34	2000	0.0002	10	110.3	115.4	92.8	129.3	97.5	80.4	20.3	21.7	17.5	24.2	17.9	14.7	—	—
36	2000	0.0002	2	37.9	—	35.0	—	35.4	—	7.2	—	6.5	—	6.5	—	6.5	—

(a) Specimen missing at end of test.

TABLE XII
 EFFECT OF CASCADE POSITION ON METAL WEIGHT LOSS OF SUPERALLOY TEST SPECIMENS

Run No.	Gas Temp., F	Fuel Sulfur, Wt. %	"Sea Salt", D ₅₀	Weight Loss, Mg. per cm ²						Per Cent Metal Weight Loss			
				Stage 1		Stage 2		Stage 3		Stage 1		Stage 2	
				1	2	3	4	5	6	1	2	3	4
35	1500	0.40	10	57.0	62.5	116.0	138.4	130.3	57.2	9.9	10.8	20.2	24.2
39	1500	0.40	10	44.6	46.8	112.0	389.9	225.1	141.2	7.8	8.2	19.6	68.1
41	1500	0.40	10	14.7	96.4	282.9	200.0	388.2	103.2	2.6	16.8	49.2	34.7
37	1500	0.40	2	0.7	0.8	0.3	0.9	0.8	0.8	0.1	0.2	0.0	0.2
36	2000	0.0002	2	—	32.7	—	44.5	—	23.5	—	5.7	—	7.8
38	2000	0.0002	10	—	59.7	—	57.7	—	37.7	—	10.5	—	10.2
34	2000	0.0002	10	110.3	115.4	92.8	129.3	97.5	80.4	20.3	21.7	17.5	24.2
36	2000	0.0002	2	37.9	—	35.0	—	35.4	—	7.2	—	6.5	—
38	2000	0.0002	10	87.7	—	36.2	—	(a)	—	16.1	—	16.0	—

(a) Specimen missing at end of test.

Analyses of metal loss data for Hastelloy R-235 at 1500 F exhaust gas temperature with 0.40 weight per cent fuel sulfur and 10 ppm "sea salt" in combustor air are shown in Appendix II. It is concluded that repeatability of metal loss varies with the magnitude of the loss; however, by using logarithms of metal loss this is changed to percentage variance which permits the use of conventional statistical methods for interpreting the data. The improvement or more uniform variance obtained by the use of logarithms may be observed by comparing the confidence intervals for the data shown graphically in Figures 14 and 15.

Hot corrosion with six Hastelloy R-235 specimens tested at a reduced "sea salt" ingestion rate (2.0 vs 10.0 ppm) is reduced to less than 1.0 mg/cm^2 for all specimens. Directionally repeatability is improved as "sea salt" ingestion rate is reduced (57.1 vs 86.4 per cent). (See Appendix II)

With six Udimet 700 test specimen in a cascade exposed to 2000 F exhaust gas temperature with 0.0002 per cent fuel sulfur and 10 ppm "sea salt" repeatability as measured by coefficient of variation is 17.1 per cent. (See Appendix II) Metal loss values for this test are shown in Figure 16.

From an examination of test specimens, the amount of scale deposit on the specimens appears to increase with an increase in "sea salt" ingestion rate at 1500 F gas temperature. At gas temperatures above approximately 1700 F, the amount of deposit accumulated on the test specimens decreased. This coincides with test specimen metal temperatures above the melting point of sodium sulfate (1623 F). Generally the nature of the attack seems to be more uniform at the higher temperatures. This probably accounts for the improved repeatability of results at 2000 F gas temperature over those at 1500 F.

7. Mass Effect of Fuel Sulfur and "Sea Salt".

An examination of data from the exploratory programs where the total mass of fuel sulfur and "sea salt" ingested by the combustor varied as operating

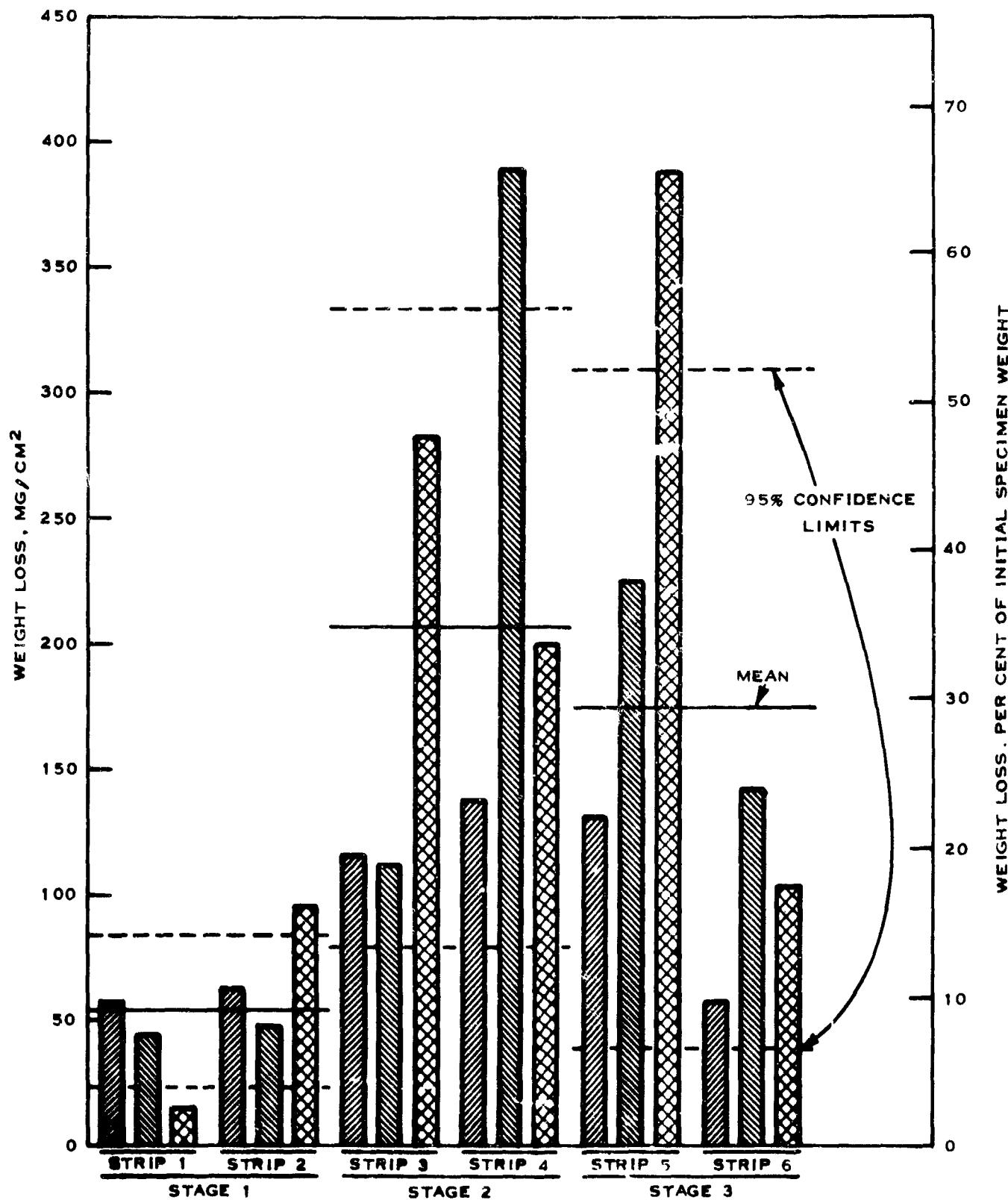


FIGURE 14
EFFECT OF CASCADE POSITION AND REPEATABILITY ON
CORROSION OF HASTELLOY R-235 AT 1500 F GAS TEMPERATURE
WITH 0.40 WT PER CENT FUEL SULFUR IN MARINE
ENVIRONMENT

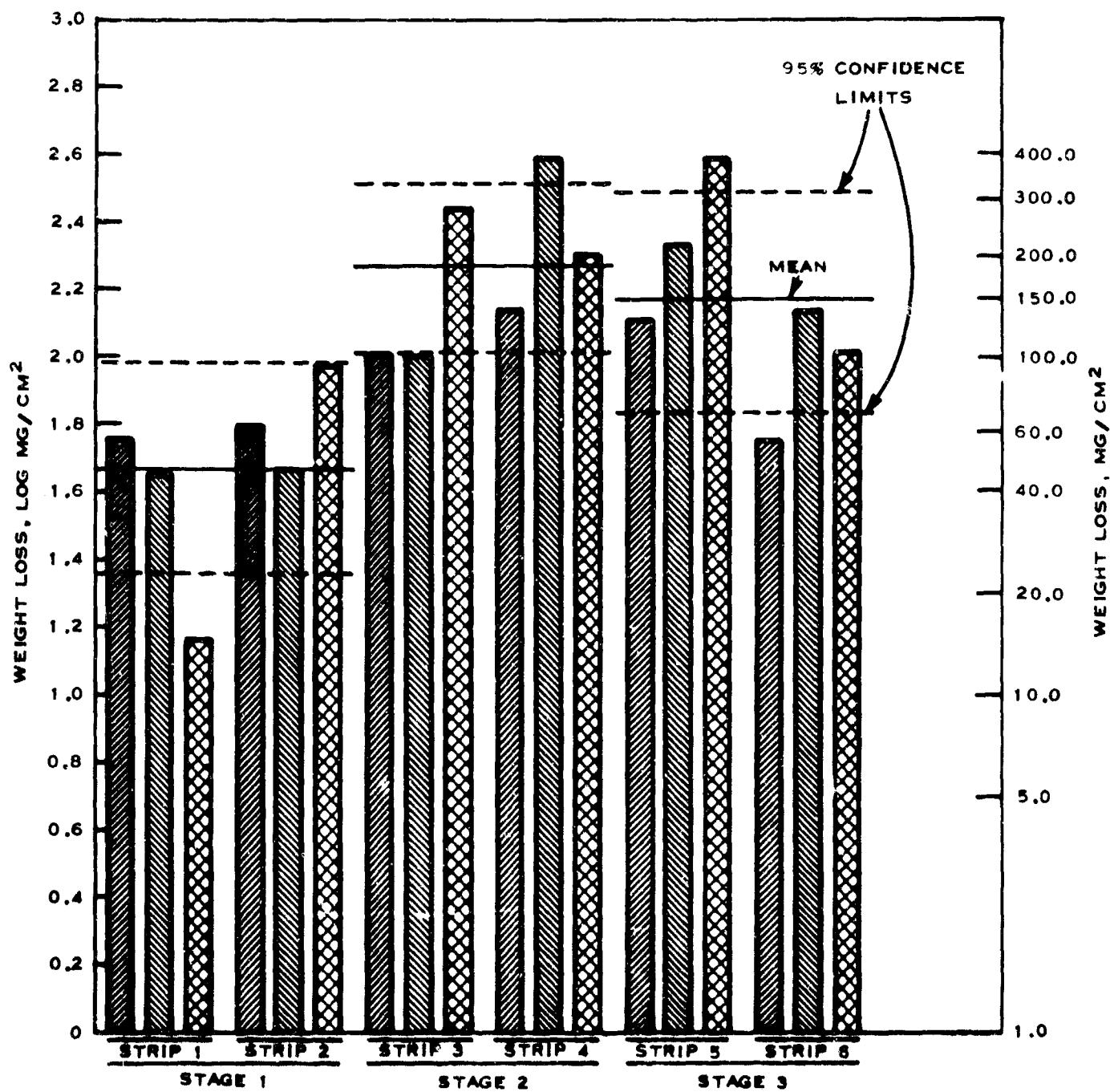


FIGURE 15
EFFECT OF CASCADE POSITION AND REPEATABILITY ON CORROSION
OF HASTELLOY R-235 AT 1500 F GAS TEMPERATURE WITH
0.40 WT. PER CENT FUEL SULFUR IN MARINE ENVIRONMENT

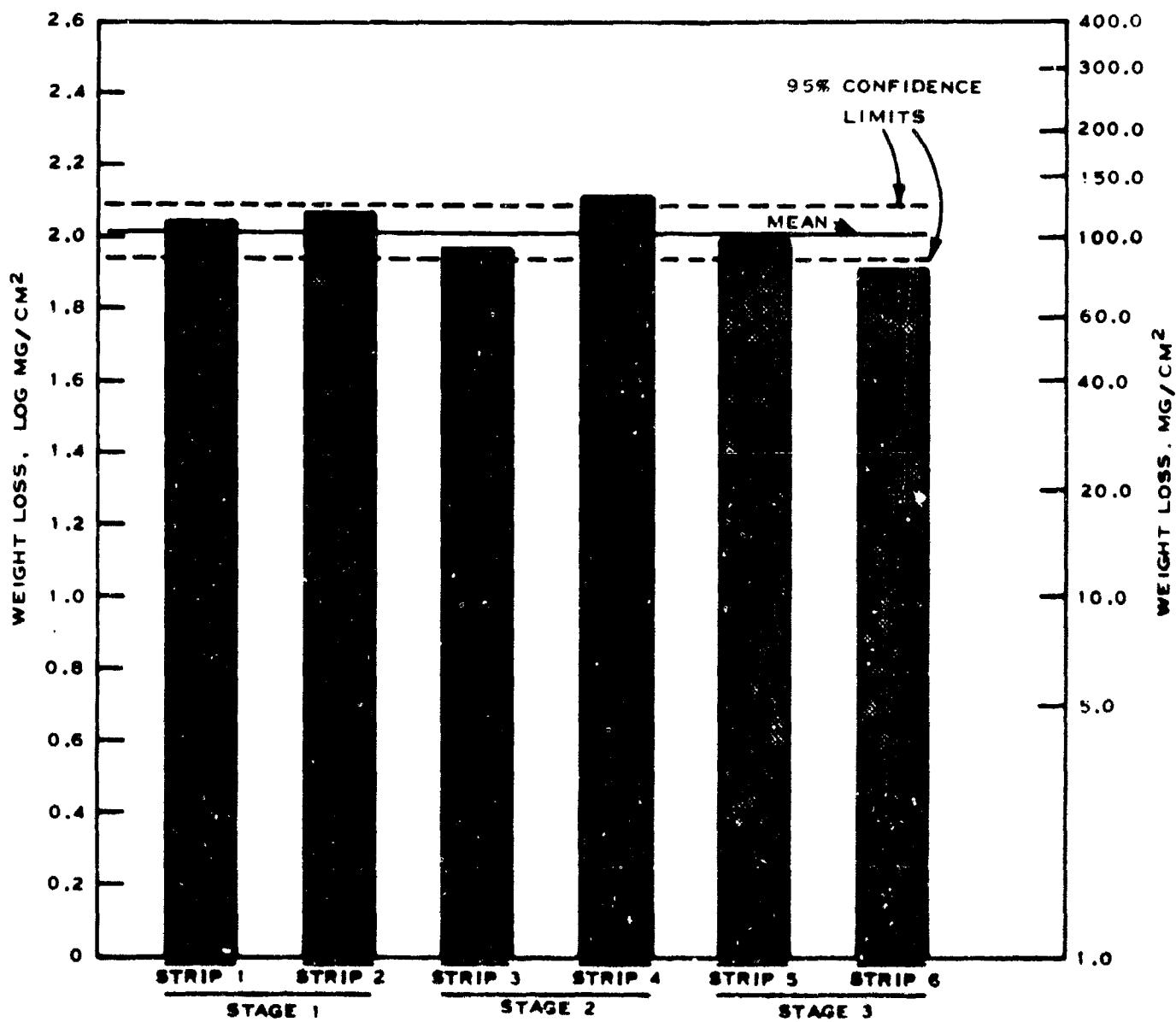


FIGURE 16
EFFECT OF CASCADE POSITION ON CORROSION OF UDIMET 700
AT 2000 F GAS TEMPERATURE WITH 0.0002 WT. PER CENT FUEL
SULFUR IN MARINE ENVIRONMENT

conditions were changed suggested that hot corrosion may be a function of the total exposure of a superalloy to these materials at a given temperature. Multiple regression analyses provide a means of evaluating the possible relationship between metal loss and mass of fuel sulfur and "sea salt" ingested. Previously data were obtained on two superalloys at 2000 F exhaust gas temperature with three levels of fuel sulfur and three levels of "sea salt" (Ref. 7). Multiple regression analyses of these data have been made and are summarized in Appendix II. The multiple regression equations for Inconel 713C and Sierra Metal 200 are both significant at the 95 per cent confidence level or above and indicate that metal loss in these data may be described by the amount of fuel sulfur and "sea salt" ingested by the combustor. The measured and calculated metal loss values for the two metals are shown in Figures 17 and 18. In Figure 18 the measured and calculated values for Sierra Metal 200 obtained during the exploratory programs are shown in addition to the data used to develop the equation. In most cases the line of equality falls within the confidence interval for the measured values. These data indicate that comparisons of metal loss for any combinations of fuel sulfur and ingested "sea salt" can be made within the limits selected for sulfur and "sea salt".

B. X-Ray Diffraction Analysis.

X-ray diffraction analyses were obtained of representative samples of scale from test specimens at various test conditions used in these exploratory programs. The scale deposit was scraped from the test specimen and then powdered prior to analysis and therefore represents an average composition. Metal loss and scale compositions for comparisons of the effect of operating conditions are shown in Tables XII through XVII. The presence of a given material is indicated as very weak, weak, medium or strong. These notations are specific to each individual material; however, they do not permit a comparison of the relative

$$\text{CALCULATED LOG MG/CM}^2 = 0.32023X_1 + 12.00854X_2 - 0.14101X_3 + 0.1183R$$

$$\text{WHERE } X_1 = \log \left[\left(\frac{\text{LBS. SULFUR}}{5 \text{ HR.}} \right) + 1.0 \right]$$

$$X_2 = \log \left[\left(\frac{\text{LBS. SEA SALT}}{5 \text{ HR.}} \right) + 1.0 \right]$$

$$X_3 = \log \left[\left(\frac{\text{LBS. SEA SALT}}{\text{LBS. SULFUR}} \right) + 1.0 \right]$$

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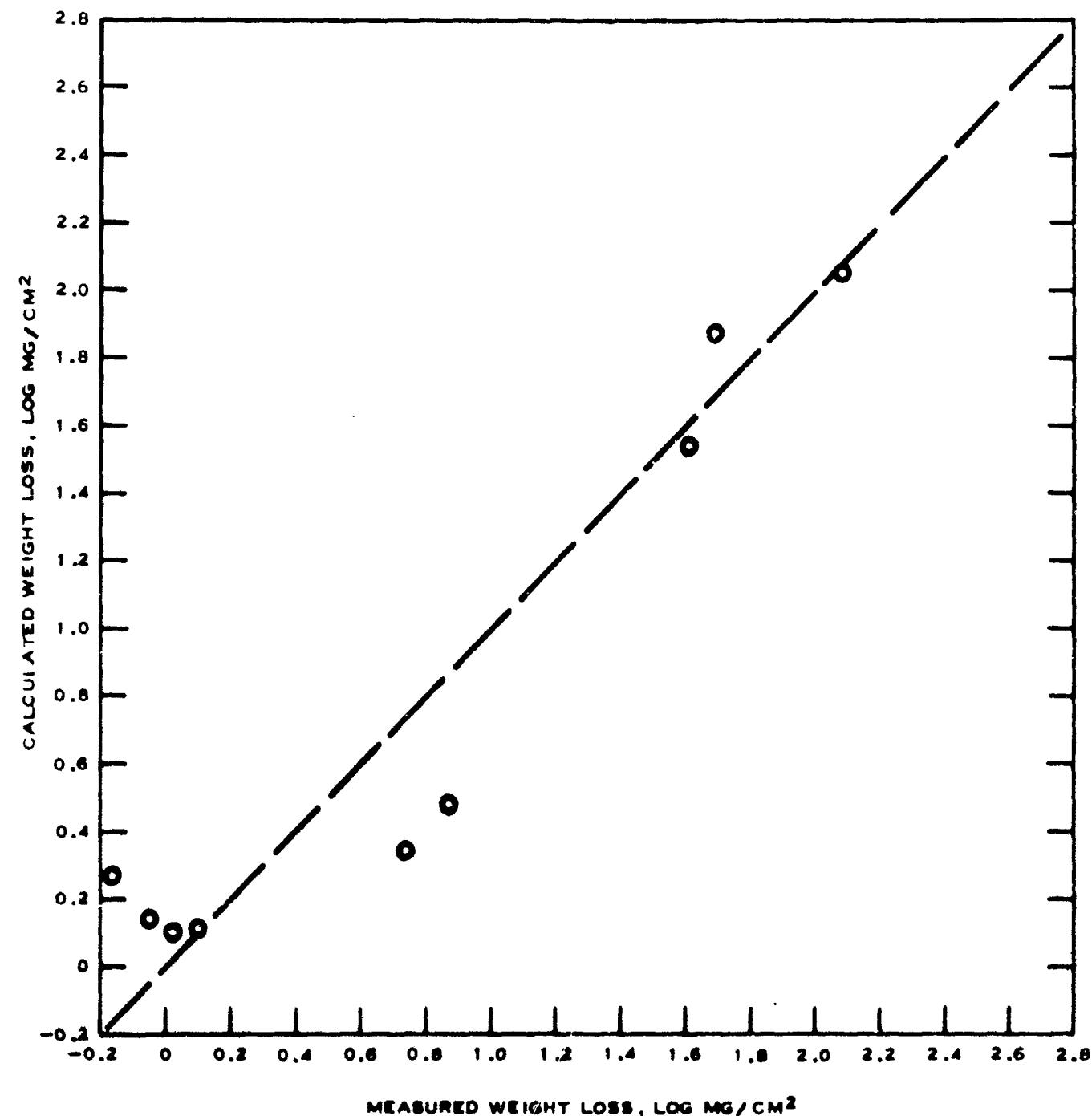


FIGURE 17
RELATIONSHIP BETWEEN MEASURED AND CALCULATED WEIGHT LOSS FOR INCONEL 713C AT 2000 F

RESEARCH DIVISION REPORT 3945-64R

$$\text{CALCULATED LOG MG/CM}^2 = -0.83899X_1 + 3.50688X_2 + 0.25689X_3 + 1.33001$$

$$\text{WHERE } X_1 = \log \left[\left(\frac{\text{LBS. SULFUR}}{5 \text{ HR.}} \right) + 1.0 \right]$$

$$X_2 = \log \left[\left(\frac{\text{LBS SEA SALT}}{5 \text{ HR.}} \right) + 1.0 \right]$$

$$X_3 = \log \left[\left(\frac{\text{LBS SEA SALT}}{\text{LBS SULFUR}} \right) + 1.0 \right]$$

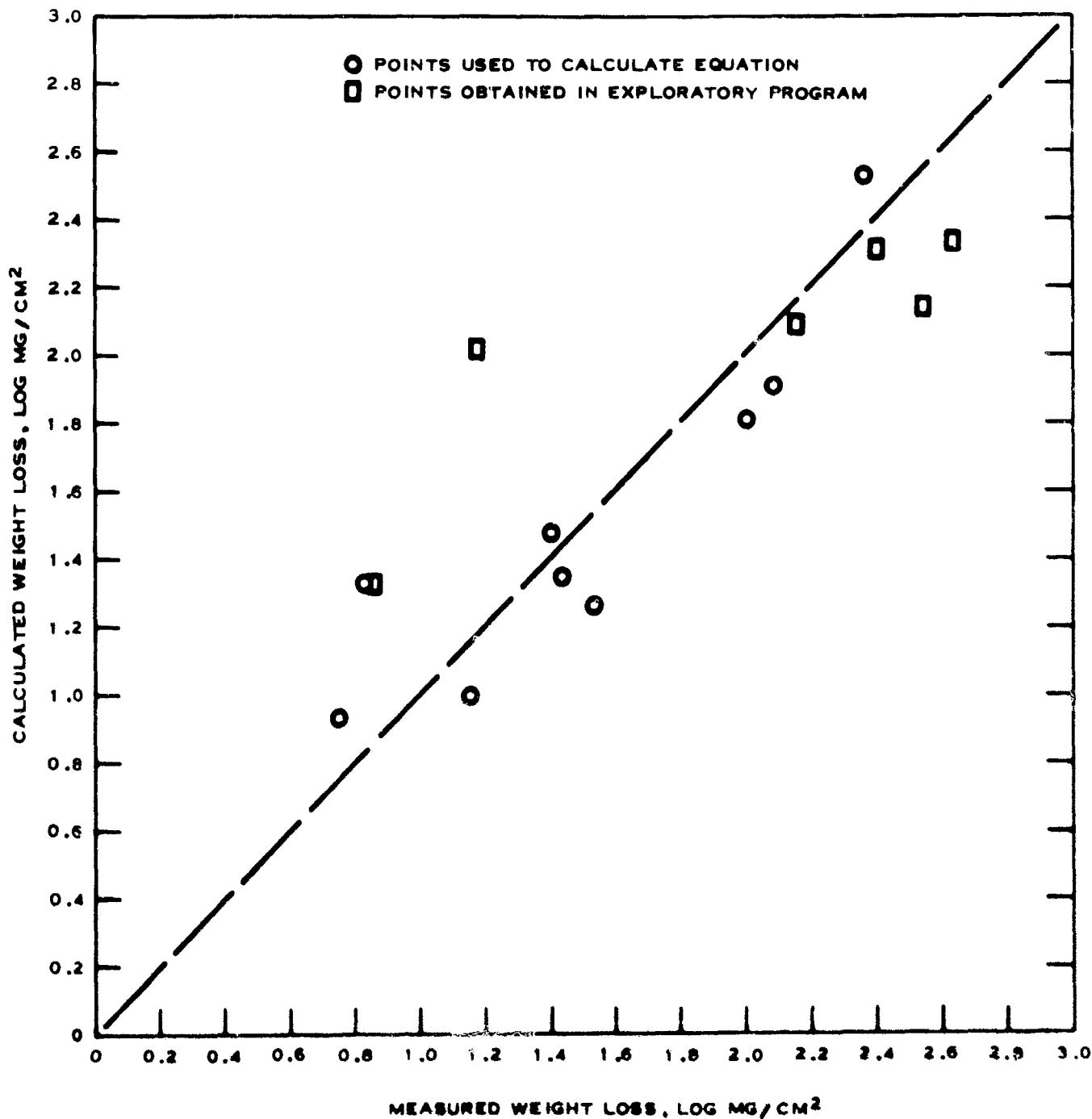


FIGURE 18
RELATIONSHIP BETWEEN MEASURED AND CALCULATED WEIGHT LOSS FOR SM200 AT 2000 F

amounts of material present in the deposit (e.g., NiO vs MgO). These data do not show relationships between operating conditions and scale composition; however, there appears to be a relationship between scale composition and metal loss. These data are summarised in Table XVIII for each of the three superalloys. Three materials identified in the scale (Periclase, Thenardite and Halite) are present as a result of "sea salt" ingestion while the lining materials are from metal corrosion. As metal loss increases the intensities of the materials from "sea salt" decrease while the intensities for those from corrosion increase. These data show no other apparent pattern with variations in operating conditions, indicating that the composition of the corrosion products from a given superalloy do not vary significantly. This suggests that variations in the extent of corrosion with changes in operating conditions are related to that phase of the attack dealing with the initial penetration of the protective oxide film, rather than with the corrosive reactions once penetration has been accomplished.

TABLE XIII
INFLUENCE OF GAS TEMPERATURE ON COMPOSITION OF DECOMPOSITES

Run No.	Gas Temp., °C.	Fuel Sulfur, wt.-%	Gas Velocity ft./sec.	Cascade Stage	Benzene HIC	Periclane MgO	Thomarite Mg ₂ SiO ₅	Halite NaCl	Nickel (II) Cr ₂ O ₃	Aluminian Chromite MgO·Cr ₂ O ₃	Nickel Ni ₃ Al	Tungstate Na ₂ WO ₄	Sodium Loss wt./cm. ²
W	W ²	W ³	W ⁴	W ⁵	W ⁶								
14	1600	C-CaO ₂	410	1	3	N	—	—	W?	W?	—	—	58.5
24	2000	0.0002	500	1	5	—	—	—	W?	W?	—	—	112.8
13	1600	0.40	410	1	W?	—	—	—	—	—	—	—	1.7
7	2000	0.40	490	1	—	—	—	—	—	—	—	—	5.5
14	1600	C-0.0002	410	2	5	—	—	—	W?	W?	—	—	169.2
8	2000	0.00002	490	2	5	—	—	—	W?	W?	—	—	72.9
15-19-41	1500	C-0.0	500	2	5	—	—	—	W?	W?	—	—	206.5
7	2000	0.40	490	2	W	—	—	—	—	—	—	—	9.5
14	1600	C-0.0002	410	3	W	—	—	—	—	—	W	—	169.2
26	2000	0.0002	500	3	5	—	—	—	W?	W?	—	—	252.1
27	1500	C-0.0	500	3	W	—	—	—	W	W	—	—	125.6
7	2000	0.40	490	3	—	—	—	—	—	—	—	—	7.0

Relative Intensities 3 = Strong W = Medium W = Weak W = Very Weak

W = Pattern not positively identified

Note: Tests made at 15 atm. combustor pressure with 10 ppm "sea salt" concentration in combustor air.

TABLE XIV
EFFECT OF COMBUSTOR PRESSURE ON COMPOSITION OF DEPOSITS

Run No.	Gas Temp., F.	Fuel Sulfur, wt. %	Combustor Pressure, Atm.	Cascade Stage		Periclase MgO	Thomardite Mg ₂ SO ₄	Halite NaCl	Nickel (III) Chromite NiO·Cr ₂ O ₄	Sodium Tungstate Na ₂ WO ₄	Weight Loss kg/cm ²
				5	15						
35-39-4.1	1500	0.40	0.40	5	2	N	W	W	—	—	12.4
	1500	0.40	0.40	15	2	S	W	W	—	—	206.5
25	1500	0.40	0.40	5	3	—	N	W	—	—	3.6
27	1500	0.40	0.40	15	3	N	W	W	—	—	125.6
26	2000	0.0002	0.0002	5	3	S	—	—	S	W?	143.5
28	2000	0.0002	0.0002	15	3	S	—	—	W	W	252.1
26	2000	0.0002	0.0002	5	1	S	W	—	W	W?	41.8
34	2000	0.0002	0.0002	15	1	S	—	—	W	W?	112.8

Relative Intensities S = Strong N = Medium W = Weak VW = Very Weak ? = pattern not positively identified

NOTE: Tests made at 10 ppm "sea salt" concentration in combustor air with a gas velocity of 500 ft./sec. at test specimen.

TABLE XV
EFFECT OF GAS VELOCITY ON COMPOSITION OF DEPOSITS

Run No.	Gas Temp. F	Fuel	Gas Velocity ft./sec.	Cascade Stage	Bunsenite	Periclaase	Thomardite	Halite	Nickel (II) Chromite NiO-Cr ₂ O ₃	Aluminian Chromite FeO-(Al ₂ Cr ₂ O ₇) ₂	Sodium Tungstate Na ₂ WO ₄	Wolfram Cr ₂ O ₃ kg/cm ²
35-39-31	1500	0.740	50	2	W	W	W	W	W	W	W	1.2
23	1500	0.740	500	2	S	W	W	W	W	W	W	206.5
27	1500	0.40	50	3	W	W	W	W	W	W	W	8.0
24	2000	0.0002	50	3	W	W	W	W	W	W	W	125.6
28	2000	0.0002	500	3	S	W	W	W	W	W	W	15.1
					S	W	W	W	W	W	W	252.1
					Stainless Steel 200	W	W	W	W	W	W	
					Hastelloy R-235	W	W	W	W	W	W	
					Udinet 700	W	W	W	W	W	W	
34	2000	0.0002	50	1	W	W	W	W	W	W	W	3.3
					Udinet 700	W	W	W	W	W	W	112.8

Relative Intensities S = Strong W = Medium W = Weak VW = Very Weak ? = pattern not positively identified

NOTE: Tests made at 10 ppm "sea salt" concentration in combustor air at 15 atm. combustor pressure.

TABLE XVI
EFFECT OF "SEA SALT" CONCENTRATION ON COMPOSITION OF DEPOSITS

Run No.	Gas Temp., F.	Psi. Sulfur, Wt. %	Sea Salt, Ppm.	Cascade Stage	Bunsenite NiO	Periclase MgO	Thomardite Na ₂ SO ₄	Halite NaCl	NiO·Cr ₂ O ₃	NiO·Cr ₂ O ₃	Nickel (II) FeC (AlCr) ₂ O ₃	Aluminian Chromite	Weight Loss %/cm ²
35-39-41	1500	0.40	10	2	S	W	W	VW	S	—	—	—	206.5
37	1500	0.40	2	2	—	W	S	W	—	—	—	—	0.6
34	2000	0.0002	10	1	S	—	—	—	—	—	—	—	—
36	2000	0.0002	2	1-2-3	S	W	—	W	W	W?	W?	W?	112.8
													36.1

Relative Intensities S = Strong M = Medium W = Weak VW = Very Weak ? = pattern not positively identified

NOTE: Tests made at 15 atm. combustor pressure with a gas velocity of 500 ft./sec. at test specimen.

TABLE VII
EFFECT OF CASCADE POSITION ON COMPOSITION OF DEPOSIT

Run No.	Gas Temp., F.	Prec. Sulfur, Mt. %	"Sea Salt," Mt. %	Cascade Stage	Bunsenite NiO	Periclase MgO	Thoenardite Na_2SO_4	Nalite NaCl	Nickel (II) Chromite $\text{NiO}\cdot\text{Cr}_2\text{O}_3$	Aluminian Chromite $\text{FeO}\cdot(\text{AlCr})_2\text{O}_3$	Weight Loss, %
35-39-41	1500	0.40	10	1	S	W	W	W	S	—	53.7
35-39-41	1500	0.40	10	2	S	W	W	W	S	—	206.5
35-39-41	1500	0.40	10	3	S	W	W	W	S	—	174.2
34	2000	0.0002	10	1	S	—	—	—	W	W?	112.8
34	2000	0.0002	10	3	S	—	—	—	W	W?	89.0

Relative Intensities S = Strong M = Medium W = Weak VV = Very Weak ? = pattern not positively identified
 NOTE: Tests made at 15 atm. combustor pressure with a gas velocity of 500 ft./sec. at test specimens.

TABLE XVIII
COMPARISON OF METAL LOSS AND SCALE COMPOSITION

C. Metallography

Intercrystalline corrosion can be identified by measurement of loss in tensile properties or by metallographic examination. The latter was used during this exploratory program because it provided additional information about changes in the metal surface resulting from exposure to corrosive agents.

Test specimens were cleaned by electrolytic descaling in molten sodium hydroxide after exposure in Phillips 2-inch combustor exhaust. This allowed for measurements of metal loss by the test specimens from hot corrosion without destruction of the surface layers where corrosion products had penetrated the metal matrix.

The photomicrographs shown in Figures 19 through 25 were taken of various superalloys after their exposure to hot corrosion in a marine environment. They are typical of the photomicrographs obtained, and illustrate the nature of the attack experienced by the superalloys over the range of conditions investigated in this exploratory program. However, it is pertinent to point out that the selection of a typical area of corrosive attack from a detailed microscopic examination of the test specimen is a difficult task. Widely divergent results are in evidence under supposedly comparable conditions, because penetration of the protective oxide surface film and erosion of the corrosion products are randomly occurring processes. Nevertheless, it is felt that the general mode of corrosive attack is clearly evident in these photomicrographs.

Figure 19 shows X250 and X1000 magnification photomicrographs of polished unetched sections of Sierra Metal 200 specimens following exposure with 10 ppm "sea salt" in air and 0.0002 weight per cent sulfur in fuel at two temperatures, 1600 F and 2000 F. The test specimen weight losses obtained

EFFECT OF GAS TEMPERATURE AT TEST SPECIMENS

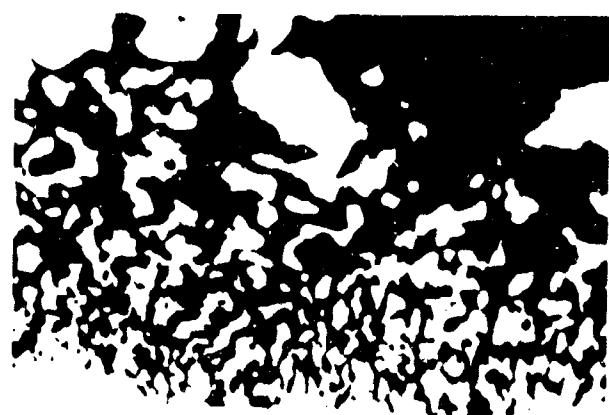
1600 F

92 MG/CM² METAL LOSS

2000 F

252 MG/CM² METAL LOSS

250X MAGNIFICATION, POLISHED, UNETCHED



1000X MAGNIFICATION, POLISHED, UNETCHED

FIGURE 19
PHOTOMICROGRAPHS OF SIERRA METAL 200 TEST SPECIMENS
AFTER EXPOSURE TO HOT CORROSION IN PHILLIPS 2-INCH COMBUSTOR OUTLET
AT TEST CONDITIONS 14 AND 28

EFFECT OF SULFUR CONCENTRATION IN FUEL

0.0002 WEIGHT PER CENT

169 MG/CM² METAL LOSS

0.40 WEIGHT PER CENT

200 MG/CM² METAL LOSS

250X MAGNIFICATION, POLISHED, UNETCHED



1000X MAGNIFICATION, POLISHED, UNETCHED

FIGURE 20
PHOTOMICROGRAPHS OF HASTELLOY R-235 TEST SPECIMENS
AFTER EXPOSURE TO HOT CORROSION IN PHILLIPS 2-INCH COMBUSTOR OUTLET
AT 1600 F TEST CONDITIONS 14 AND 41

EFFECT OF SULFUR CONCENTRATION IN FUEL

0.0002 WEIGHT PER CENT

73 MG/CM² METAL LOSS

0.40 WEIGHT PER CENT

10 MG/CM² METAL LOSS

250X MAGNIFICATION, POLISHED, UNETCHED

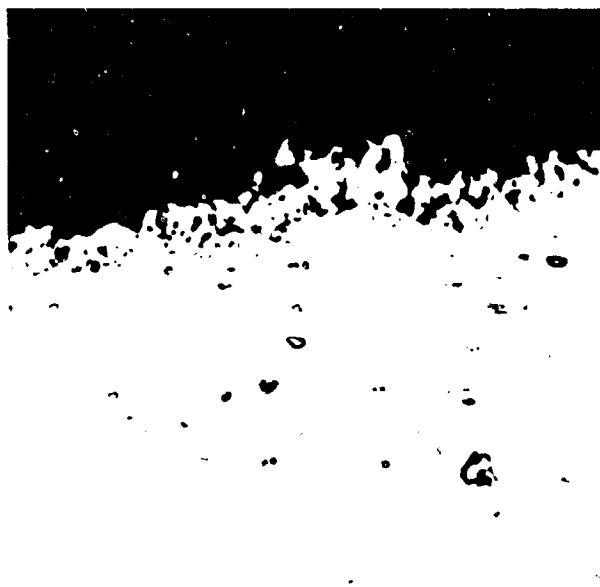


1000X MAGNIFICATION, POLISHED, UNETCHED

FIGURE 21
PHOTOMICROGRAPHS OF HASTELLOY R-235 TEST SPECIMENS
AFTER EXPOSURE TO HOT CORROSION IN PHILLIPS 2-INCH COMBUSTOR OUTLET
AT 2000 F TEST CONDITIONS 7 AND 8

EFFECT OF GAS PRESSURE AT TEST SPECIMENS

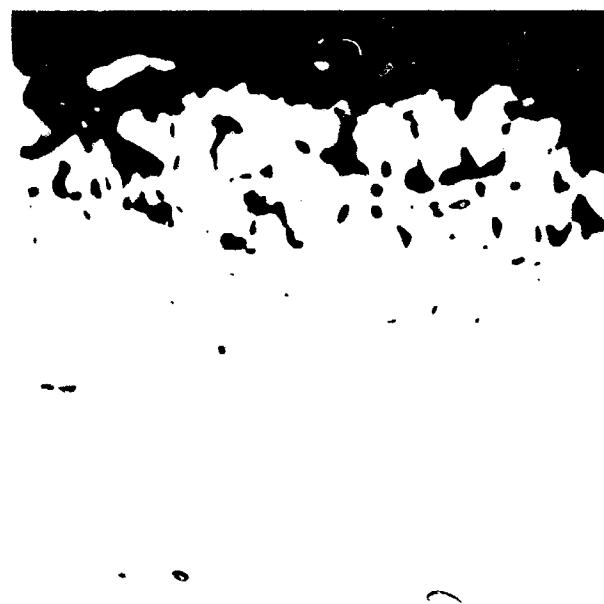
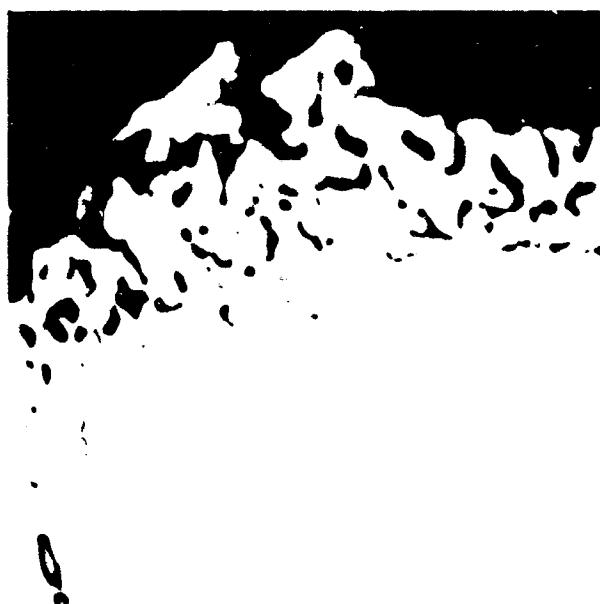
5 ATMOSPHERES

12 MG/CM² METAL LOSS

15 ATMOSPHERES

200 MG/CM² METAL LOSS

250X MAGNIFICATION, POLISHED, UNETCHED



1000X MAGNIFICATION, POLISHED, UNETCHED

FIGURE 22

PHOTOMICROGRAPHS OF HASTELLOY R-235 TEST SPECIMENS
AFTER EXPOSURE TO HOT CORROSION IN PHILLIPS 2-INCH COMBUSTOR OUTLET
AT 1500 F TEST CONDITIONS 25 AND 41



EFFECT OF GAS VELOCITY AT TEST SPECIMENS

50 FEET PER SECOND

8 MG/CM² METAL LOSS

500 FEET PER SECOND

126 MG/CM² METAL LOSS

250X MAGNIFICATION, POLISHED, UNETCHED



1000X MAGNIFICATION, POLISHED, UNETCHED

FIGURE 23

PHOTOMICROGRAPHS OF SIERRA METAL 200 TEST SPECIMENS
AFTER EXPOSURE TO HOT CORROSION IN PHILLIPS 2-INCH COMBUSTOR OUTLET
AT 1500 F TEST CONDITIONS 23 AND 27

EFFECT OF SEA SALT CONCENTRATION IN AIR

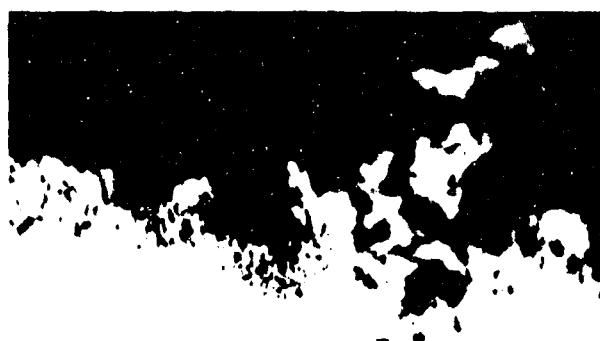
2 PARTS PER MILLION

38 MG/CM² METAL LOSS

10 PARTS PER MILLION

98 MG/CM² METAL LOSS

250X MAGNIFICATION, POLISHED, UNETCHED



1000X MAGNIFICATION, POLISHED, UNETCHED

FIGURE 24
 PHOTOMICROGRAPHS OF UDIMET 700 TEST SPECIMENS
 AFTER EXPOSURE TO HOT CORROSION IN PHILLIPS 2-INCH COMBUSTOR OUTLET
 AT 2000 F TEST CONDITIONS 34 AND 36

EFFECT OF TEST SPECIMEN POSITION IN CASCADE

STAGE 1

47 MG/CM² METAL LOSS

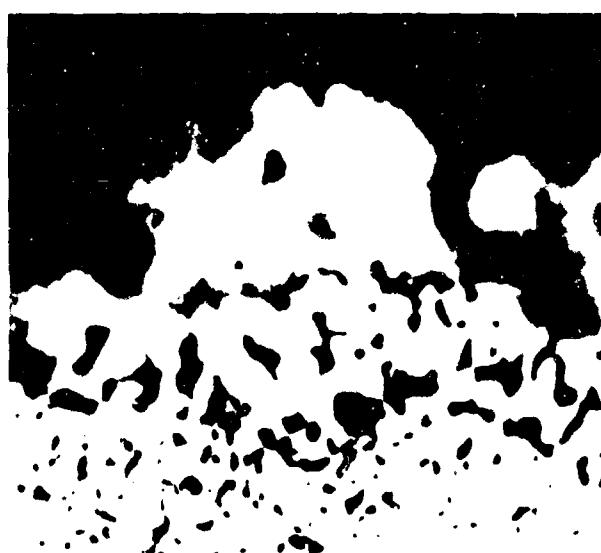


STAGE 3

103 MG/CM² METAL LOSS



250X MAGNIFICATION, POLISHED, UNETCHED



1000X MAGNIFICATION, POLISHED, UNETCHED

FIGURE 25

PHOTOMICROGRAPHS OF HASTELLOY R-235 TEST SPECIMENS
AFTER EXPOSURE TO HOT CORROSION IN PHILLIPS 2-INCH COMBUSTOR OUTLET
AT 1500 F TEST CONDITION 41

at the two different temperatures are indicated for aid in interpretation of the photomicrographs. These test specimens were chosen to show the nature of corrosive attack where the rate of metal loss increased rapidly with increasing gas temperature, as was plotted in Figure 8.

Figures 20 and 21 show X250 and X1000 magnification photomicrographs of polished unetched sections of Hastelloy R-235 specimens following exposure with 10 ppm "sea salt" in air and with both 0.0002 and 0.40 weight per cent sulfur in fuel at 1600 F and 2000 F, respectively. These test specimens were chosen to show the nature of corrosive attack over this range in gas temperature because there was a reversal in the effect of fuel sulfur content on metal loss, as was plotted in Figure 6.

Figure 22 shows X250 and X1000 magnification photomicrographs of polished unetched sections of Hastelloy R-235 specimens following exposure with 10 ppm "sea salt" in air and 0.40 weight per cent sulfur in fuel at two gas pressures, 5 and 15 atmospheres. These test specimens were chosen to show the nature of corrosive attack where the metal loss increased rapidly with increasing pressure, as was plotted in Figure 10.

Figure 23 shows X250 and X1000 magnification photomicrographs of polished unetched sections of Sierra Metal 200 specimens following exposure with 10 ppm "sea salt" in air and 0.40 weight per cent sulfur in fuel at two gas velocities, 50 and 500 feet per second. These test specimens were chosen to show the nature of corrosive attack where metal loss increased rapidly with increasing velocity, as was plotted in Figure 12.

Figure 24 shows X250 and X1000 magnification photomicrographs of polished unetched sections of Udimet 700 specimens following exposure at 2000 F with 0.0002 weight per cent sulfur in fuel and with two levels of "sea salt" in air, 2 and 10 ppm. These test specimens were chosen to show the nature of corrosive attack where the metal loss increased rapidly with increasing "sea salt" concentration, as was tabulated in Table XI.

Figure 25 shows X250 and X1000 magnification photomicrographs of polished unetched sections of Hastelloy R-235 specimens from stages 1 and 3 in the cascade following exposure with 10 ppm "sea salt" in air and 0.40 weight per cent sulfur in fuel at 1500 F. The specimen from stage 2 of this test was shown in Figure 20. These test specimens were chosen to show the nature of corrosive attack under conditions where position in the cascade had a significant effect on the severity of metal loss, as was plotted in Figure 14.

In general, photomicrographs taken at X250 magnification showed only a shallow penetration of corrosion products, despite the catastrophic rates of metal loss encountered in the presence of "sea salt". The depth of surface penetration ranged from less than one to a maximum of about four thousands of an inch. It also appeared that surface roughness increased with metal loss, probably as a result of the undermining of metal grains by intergranular attack. These observations are in agreement with previous studies which showed hot corrosion in a marine environment to result in no deep intergranular penetration of sulfur into the matrix of the superalloys and little or no loss in tensile properties. (4 and 7)

Photomicrographs, taken at X1000 magnification to show the corrosive attack in greater detail, consistently reveal penetration of the metal matrix being led by randomly dispersed light-grey globules of metallic sulfides. This mode of corrosive attack is usually referred to as sulfidation, and occurred despite the prevalence of oxidizing conditions. The formation of these sulfide particles is associated with changes in surface composition of the alloy which is characterized by chromium depletion. Rapid oxidation of the weakened layer of alloy follows sulfide penetration, at times so closely as to give the appearance of gross oxidation.

VI. CONCLUSIONS

The following statements can be made concerning the effect of fuel sulfur and operating conditions on hot corrosion of superalloys in a marine environment. They are based on exploratory programs in which specimens of six superalloys were exposed to vitiated air from the Phillips 2-inch combustor during five hour cyclic tests (55 minutes fuel-on, 5 minutes fuel-off).

1. An increase in fuel sulfur content (0.0002 to 0.40 weight per cent) at high pressure (15 atmospheres) and high velocity (330 to 510 feet per second at test specimen) with 10.0 ppm "sea salt" in combustor air over a range of temperature from 1200 to 2200 F:
 - a. Inhibited metal loss at gas temperatures of 1800 F and above.
 - b. Increased metal loss for some superalloys at 1600 F and below.
2. An increase in combustor pressure (5 to 15 atmospheres) accelerated hot corrosion for those superalloys subject to attack with 10.0 ppm "sea salt" in combustor air at both the 1500 F gas temperature with 0.40 weight per cent fuel sulfur and the 2000 F gas temperature with 0.0002 weight per cent fuel sulfur test conditions.
3. An increase in gas velocity at the test specimen increased hot corrosion for those superalloys subject to attack with 10.0 ppm "sea salt" in combustor air at both the 1500 F gas temperature with 0.40 weight per cent fuel sulfur and the 2000 F gas temperature with 0.0002 weight per cent fuel sulfur test conditions.
4. A change in air fuel ratio from 50 to 75 did not change hot corrosion for one superalloy tested at 2000 F gas temperature with 0.0002 weight per cent fuel sulfur and 10.0 ppm "sea salt" in combustor air.

5. An increase in "sea salt" ingestion rate from 2.0 to 10.0 ppm in combustor air increased hot corrosion; however, the magnitude of the increase varies with temperature-sulfur combinations or metals.
6. Cascade testing provides additional information on hot corrosion from each test:
 - a. At 2000 F gas temperature with 10.0 ppm "sea salt" in combustor air and 0.0002 weight per cent fuel sulfur, repeatability is good (17.1 per cent) and hot corrosion is uniform from stage to stage in the cascade
 - b. At 1500 F gas temperature with 0.40 weight per cent fuel sulfur and 2.0 or 10.0 ppm "sea salt" in combustor air repeatability is poorer (57.1 to 86.4 per cent) than at high temperature and severity of hot corrosion varies from stage to stage with high metal loss.
7. Hot corrosion is a function of mass exposure to "sea salt" in combustor air, fuel sulfur and the ratio of "sea salt" to fuel sulfur for two superalloys at 2000 F gas temperature.
8. Materials from "sea salt" in specimen scale deposits decrease and materials from corrosion products increase as metal loss increases.
9. Typically, the micro features of hot corrosion in a marine environment which were evident in this exploratory program were a metal surface roughened by intergranular attack with shallow sulfide penetration of the matrix preceding oxidation.

VII. RECOMMENDATIONS

This study was made to determine the effect of fuel sulfur over a range of operating conditions on hot corrosion of superalloys in a marine environment to provide information to aid in selection of operating conditions for a program designed to determine whether the maximum sulfur limit of 0.40 weight per cent, currently allowed in grade JP-5 aviation turbine fuel, is a safe level for protection of turbine blade alloys used in advanced engines.

A reversal of the effect of fuel sulfur on hot corrosion is shown between tests at 1600 F and below and 1800 F and above for some superalloys. Increasing combustor pressure and gas velocity increased metal loss for those superalloys subject to attack with no apparent reversals. An analysis of data obtained at 2000 F with two superalloys shows that hot corrosion is a function of mass exposure to fuel sulfur and "sea salt".

Therefore, it is recommended that, in a more complete investigation, tests should be conducted using:

- (1) Gas temperatures of 1800 F, 2000 F and 2200 F to give metal temperatures below (1700 F) near (1850 F) and above (2000 F) the maximum recommended operating limits for the superalloys being used should be investigated to provide an answer to the primary objective of the program.
- (2) To accelerate hot corrosion, test conditions approaching actual engine conditions (within test stand capabilities) should be used. A combustor pressure of 15 atmospheres should be used and gas velocity varied with temperature to permit mass of air and fuel to be held constant throughout the program.
- (3) Tests should also be conducted at a gas temperature of 1500 F (below the point of reversal of the effect of fuel sulfur) to provide supplemental information.

(4) A cascade test specimen holder should be used in future investigations since additional information can be obtained at a nominal increase in test costs.

VIII. FUTURE WORK

A test program to determine whether the maximum sulfur limit of 0.40 weight per cent, currently allowed in grade JP-5 aviation turbine fuel, is a safe level for the protection of turbine blade alloys in advanced engines will be conducted during the last six months of this contract. Significant points in the program are as follows:

1. Exhaust Gas Temperature. Three levels of exhaust gas temperature (1800, 2000 and 2200 F) will be used representing current and future engine designs to answer the primary objective of the program. If possible a fourth temperature (1500 F) will then be used.
2. "Sea Salt" Concentration. Three levels of "sea salt" in combustor air (zero, 1.0 and 10.0 ppm) have been selected as representing the possible range of exposure.
3. Fuel Sulfur Concentration. Three levels of fuel sulfur have been selected (0.0002, 0.040 and 0.40 weight per cent) which represent the range of values from the current specification maximum down to that obtainable by hydrotreating.
4. Combustor Pressure. A combustor pressure of 15 atmospheres will be used for all tests and has been selected as representative of future engines and also to accelerate hot corrosion.
5. Mass Exposure to Sulfur and "Sea Salt". A fuel flow of 120 pounds per hour and air flow of 7200 pounds per hour will be maintained constant throughout the test program. While this will result in variations in gas velocity with temperature, the nine combinations

5. (Cont'd) of sulfur and "sea salt" will provide a measure of the temperature effect that is not confounded with the effect of mass of sulfur and "sea salt".

6. Cascade Testing. One specimen of each of six superalloys will be mounted in a three stage cascade with two superalloys in each stage. Each stage of the cascade will be successively rotated 120 degrees to prevent channeling of hot gas flow. Each superalloy will be tested once in each stage of the cascade.

7. Superalloys. Five superalloys of interest for use as turbine blade and/or guide vane materials for engines of advanced design have been selected and their compositions are shown in Table XIX. In addition one superalloy with an aluminum diffusion coating (Inconel 713C with Misco coating No. MDC-1) has also been included in the program. Coupons of superalloys to be evaluated are 0.50 in. wide, 2.38 in. long and 0.12 in. thick. The coupons have been increased in thickness from 0.06 in. previously used to 0.12 in. to provide additional strength and prevent bending and breakage which result in lost specimens.

TABLE XIX
COMPOSITION OF SUPERALLOYS

<u>Alloying Elements</u>	<u>Nominal Composition, per cent</u>				
	<u>INCO-713C</u>	<u>SM-200</u>	<u>IN-100</u>	<u>Udimet 500</u>	<u>WI-52</u>
Nickel	72	60	60	51	--
Cobalt	--	10	15	19	63
Chromium	13	9	10	19	21
Molybdenum	4.5	--	3	4	--
Tungsten	--	12	--	--	11
Aluminum	6	5	5.5	3	--
Titanium	0.6	2	5	3	--
Iron	1	0.5	0.5	1	3
Columbium	2	1	--	--	2
Vanadium	--	--	1	--	--
Carbon	0.1	0.2	0.2	0.1	0.5

8. Evaluation. The following information will be obtained on superalloy test specimens exposed to hot gases from the Phillips 2-inch combustor:

- (a) Metal Loss measured as difference in weight of test specimens prior to test and after cathodically descaling at the end of test.
- (b) Metallography. To aid in evaluating the extent and mode of corrosive attack microscopic examination with photomicrographs will be made of test specimens.

The test plan for this program is shown in Table XX. Based on exploratory tests which indicate good repeatability at 2000 F this program has been set up as a randomized block experiment with each superalloy to be tested once in each of the three stages of the cascade at each temperature-fuel sulfur—"sea salt" combination. Statistical analyses of these data will provide evaluations of the effects, and interactions, for each of the six superalloys, of the three test variables (a) sulfur concentration in the fuel, (b) "sea salt" in combustor air, and (c) exhaust gas temperature. These data will also be examined to determine if mass of fuel sulfur and "sea salt" explain variations in hot corrosion of the superalloys.

TABLE XX
TEST PLAN FOR HCT CORROSION STUDIES

Temperature, °F	Fuel Sulfur Wt. %	Sea Salt in Air, ppm	Test Order		
			Set I	Set II	Set III
2200	0.0002	0	15	48	35
2200	0.0002	1.0	47	23	67
2200	0.0002	10.0	13	43	53
2200	0.040	0	10	40	17
2200	0.040	1.0	5	66	22
2200	0.040	10.0	65	77	7
2200	0.40	0	59	26	79
2200	0.40	1.0	31	55	8
2200	0.40	10.0	63	6	30
2000	0.0002	0	70	50	75
2000	0.0002	1.0	14	68	64
2000	0.0002	10.0	81	72	20
2000	0.040	0	74	51	18
2000	0.040	1.0	69	46	60
2000	0.040	10.0	25	21	7
2000	0.40	0	73	56	62
2000	0.40	1.0	33	37	2
2000	0.40	10.0	56	71	12
1800	0.0002	0	32	28	34
1800	0.0002	1.0	44	61	45
1800	0.0002	10.0	78	24	29
1800	0.040	0	42	27	16
1800	0.040	1.0	4	39	80
1800	0.040	10.0	38	52	11
1800	0.40	0	54	19	76
1800	0.40	1.0	49	9	41
1800	0.40	10.0	3	1	58

ARRANGEMENT OF TEST SPECIMENS

	Stage 1		Stage 2		Stage 3	
	1	2	3	4	5	6
Set I	Inco 713C	Inco 713C + MDC-1	IN-100	SM-200	U-500	WI-52
Set II	IN-100	SM-200	U-500	WI-52	Inco 713C	Inco 713C + MDC-1
Set III	U-500	WI-52	Inco 713C	Inco 713C + MDC-1	IN-100	SM-200

TABLE XX (Continued)

NOTES:

(a) Other Phillips 2-inch combustor operating variables to be held constant throughout the test program.

Combustor Inlet Air Pressure, atmosphere	15.0
Air-Fuel Ratio, pounds per pound	60
Mass Air Flow, pounds per hour	7200
Test Duration, hours	5.00

Inlet air temperature to be varied over a range of from 900 to 1200 F, as required to obtain desired exhaust gas temperature.

Thermal shock to be introduced by an operating cycle of 55 minutes at test condition, followed by 5 minutes with fuel off.

IX. ACKNOWLEDGEMENT

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- (a) Test Equipment Design and Operations by E. H. Fromm
- (b) Metallographic Analysis by H. W. Schutz,
- (c) Scale Analysis by L. V. Wilson,
- (d) Statistical Analysis by M. R. Goss and D. L. Weeks.

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APPENDIX I

TABLE XII

OPERATING CONDITIONS OF PHILLIPS 2-INCH COMBUSTOR

Test Variables Test Number	Test Conditions (a)					
	19 & 20 1200	17 & 18 1400	13 & 14 1600	15 & 16 1800	7 & 8 2000	9 & 10 2200
Calculated Gas Temperature, F						
Temperature, F						
Exhaust Gas	1195	1375	1590	1780	1990	2090 (b)
Profile (c)	20	210	135	135	150	200
Test Specimens (d)	(e)	1610(f)	1650(f)	1685	1865	1955
Combustor Inlet Air	800	800	800	1000	1000	1000
Pressure, atmospheres						
Combustor Inlet Air	15.0	15.0	15.0	15.0	15.0	15.0
Test Specimen Drop	1.4	1.5	1.6	1.7	1.7	1.5
Mass Flow Rate, pounds per hour						
Air	5400	5400	5400	5400	5400	5400
Fuel	36	54	72	72	90	108
Air Fuel Ratio	150	100	75	75	60	50
Flow Velocity, feet per second						
Combustor Reference (g)	170	170	170	200	200	200
Exhaust Gas (h)	180	195	220	240	260	275
at Test Specimens (i)	330	370	410	450	490	510
Test Duration, hours (j)	5	5	5	5	5	5
Fuel Sulfur, weight per cent	(k)	(k)	(k)	(k)	(k)	(k)
"Sea Salt", parts per million (l)	10.0	10.0	10.0	10.0	10.0	10.0

(a) Average values.

(b) Temperature at exhaust gas core averaged 2175 F but the overall lower average resulted from heat losses to water cooled wall.

(c) Maximum variation between four thermocouples on equal area centers.

(d) First pair of specimens.

(e) Below operating range of optical pyrometer.

(f) Test specimens probably reflecting flame radiation to give fictitiously high readings with optical pyrometer.

(g) Cold flow, based on 2.66 in.² exit area in flame tube.

(h) Based on 3.36 in.² area at outlet from combustor.

(i) Based on 1.80 in.² unblocked area in first test specimen holder.

(j) Operating cycle of 55 minutes at test conditions, followed by 5 minutes fuel off.

(k) 0.0002 Weight per cent for Runs Nos. 8, 9, 14, 15, 18 and 19 and 0.40 weight per cent for Run Nos. 7, 10, 13, 16, 17, 20.

(l) Based on combustor air.

TABLE XXI (Continued)

Test Variables	Test Conditions (a)					
	23 1500	24 2000	25 1500	26 2000	27 1500	28 2000
Test Number						
Calculated Gas Temperature, F						
Temperature, F						
Exhaust Gas	1410	2010	1450	1900	1495	1990
Profile (b)	40	325	100	120	225	150
Test Specimen (c)	1490	1660	--	1750	1625	1730
Combustor Inlet Air	800	800	1100	1000	800	800
Pressure, atmospheres						
Combustor Inlet Air	15	15	5	5	15	15
Test Specimen Drop	0.03	0.03	0.95	0.63	1.7+	1.7+
Mass Flow Rate, pounds per hour						
Air	685	685	2270	1800	6840	5400
Fuel	8.6	10.8	15.4	32.0	81.0	108
Air Fuel Ratio	80	63	147	56	84	50
Flow Velocity, feet per second						
Combustor Reference (d)	22	22	268	199	218	172
Exhaust Gas (e)	26	34	260	255	267	264
at Test Specimen (f)	48	63	485	476	499	494
Test Duration, hours (g)	5	5	5	5	5	5
Fuel Sulfur, weight per cent	0.40	0.0002	0.40	0.0002	0.40	0.0002
"Sea Salt", parts per million (h)	10.0	10.0	10.0	10.0	10.0	10.0

(a) Average values.

(b) Maximum variation between four thermocouples on equal area centers.

(c) First pair of specimens.

(d) Cold flow, based on 2.66 in.² exit area in flame tube.(e) Based on 3.36 in.² area at outlet from combustor.(f) Based on 1.80 in.² unblocked area in first test specimen holder.

(g) Operating cycle of 55 minutes at test conditions, followed by 5 minutes fuel off.

(h) Based on combustor air.

TABLE XII (Continued)

Test Variables Test Number	Test Conditions (a)					
	31 2000	32 2000	33 1500	34 2000	35 1500	36 2000
Calculated Gas Temperature, F						
Temperature, F						
Exhaust Gas	1960	1890	1470	1880	1500	1910
Profile (b)	345	180	225	315	195	420
Test Specimen (c)	1760	1745	1690	1730	1660	1640
Combustor Inlet Air	800	800	800	800	800	800
Pressure, atmospheres						
Combustor Inlet Air	15 1.7+	15 0.43	15 0.66	15 1.48	15 1.7+	15 1.46
Mass Flow Rate, pounds per hour						
Air	7130	2700	3420	5400	6840	5400
Fuel	144	54	40	108	81	108
Air Fuel Ratio	50	50	84	50	84	50
Flow Velocity, feet per second						
Combustor Reference (d)	227	86	109	172	218	172
Exhaust Gas (e) at Test Specimen (f)	345 644	127 237	132 246	253 472	268 500	256 478
Test Duration, hours (g)	5	5	5	5	5	5
Fuel Sulfur, weight per cent	0.0002	0.0002	0.40	0.0002	0.40	0.0002
"Sea Salt", parts per million (h)	10.0	10.0	10.0	10.0	10.0	2.0

(a) Average values.

(b) Maximum variation between four thermocouples on equal area centers.

(c) First pair of specimens.

(d) Cold flow, based on 2.66 in.² exit area in flame tube.

(e) Based on 3.36 in.² area at outlet from combustor.

(f) Based on 1.80 in.² unblocked area in first test specimen holder.

(g) Operating cycle of 55 minutes at test conditions, followed by 5 minutes fuel off.

(h) Based on combustor air.

TABLE XXI (Continued)

Test Variable Test Number	Test Conditions (a)			
	37 1500	38 2000	39 1500	41 1500
Calculated Gas Temperature, F				
Temperature, F				
Exhaust Gas	1485	1915	1520	1510
Profile (b)	200	265	170	240
Test Specimen (c)	1600	1850	1610	1675
Combustor Inlet Air	800	1200	800	800
Pressure, atmospheres				
Combustor Inlet Air	15 1.7+	15 1.52	15 1.7+	15 1.7+
Test Specimen Drop				
Mass Flow Rate, pounds per hour				
Air	6840	5400	6840	6840
Fuel	81	72	81	81
Air Fuel Ratio	84	75	84	84
Flow Velocity, feet per second				
Combustor Reference (d)	218	226	218	218
Exhaust Gas (e) at Test Specimen (f)	266 497	256 479	271 506	270 503
Test Duration, hours (g)	5	4½	5	5
Fuel Sulfur, weight per cent	0.40	0.0002	0.40	0.40
"Sea Salt", parts per million (h)	2.0	10.0	10.0	10.0

(a) Average values.

(b) Maximum variations between four thermocouples on equal area centers.

(c) First pair of specimens.

(d) Cold flow, based on 2.66 in.² exit area in flame tube.

(e) Based on 3.36 in.² area at outlet from combustor.

(f) Based on 1.80 in.² unblocked area in first test specimen holder.

(g) Operating cycle of 55 minutes at test conditions, followed by 5 minutes fuel off.

(h) Based on combustor air.

APPENDIX II

STATISTICAL ANALYSIS OF DATA

1. Repeatability of Metal Loss.

In previous studies (Ref. 7) using two superalloys it was found that variability of metal loss was a function of the magnitude of the loss. In this situation conventional statistical methods should not be used to interpret the data; however, by using a transformation of the data to provide uniform variance statistical methods may be used. In cases where variability is a function of the magnitude of the values a logarithmic transformation of the data will convert variance to a percentage value and will satisfy the requirements of uniform variance.

To evaluate the repeatability of metal loss using a cascade system for mounting test specimens, three tests were conducted using Hastelloy R-235 at 1500 F gas temperature with 0.40 weight per cent fuel sulfur and 10 ppm "sea salt" in combustor air. Metal loss per unit area for the test specimens are shown in Table XXII. Also shown are the variance, standard deviation and coefficient of variation calculated for each stage of the cascade.

APPENDIX II (Continued)

TABLE XXII

REPEATABILITY OF METAL LOSS (MG/CM²) FOR HASTELLOY R-235^(a)

Run	Weight Loss, Mg/Cm ²					
	Stage 1		Stage 2		Stage 3	
	Strip 1	Strip 2	Strip 3	Strip 4	Strip 5	Strip 6
35	57.0	62.5	116.0	138.4	130.3	57.2
39	44.6	46.8	112.0	389.9	225.1	141.2
41	14.7	96.4	282.9	200.0	388.2	103.2
TOTAL	322.0		1239.2		1045.2	
Mean	53.7		206.5		174.2	
M.S. (b)	712.61		12,254.57		14,026.60	
S.D. (c)	26.7		110.7		118.4	
C.V., % (d)	49.8		53.6		68.0	

(a) 1500 F gas temperature, 0.40 wt. per cent fuel sulfur, 10 ppm "sea salt" in combustor air.

(b) Mean square (Variance).

(c) Standard deviation.

(d) Coefficient of variation. $\left[(S.D./Mean) (100) \right]$

The standard deviations calculated for the three stages differ in size while the coefficients of variation are of the same relative magnitude. Using Bartlett's test of homogeneity of variance (Ref. 11) it can be concluded that differences in variance for the three stages are larger than would be expected by chance. Since the variances differ while the coefficients of variation are of the same magnitude the use of logarithms of the weight loss data is indicated. Logarithms of metal loss per unit area are shown in Table XXIII along with the geometric mean weight loss, variance, standard deviation and coefficient of variation for each stage of the cascade.

APPENDIX II (Continued)

TABLE XXII

REPEATABILITY OF METAL LOSS (LOG MG/CM²) FOR HASTELLOY R-235^(a)

Run	Weight Loss, Log mg/cm ²						<u>Total</u>
	<u>Stage 1</u>	<u>Stage 2</u>	<u>Stage 3</u>	<u>Strip 1</u>	<u>Strip 2</u>	<u>Strip 3</u>	
35	1.75587	1.79588	2.06446	2.14114	2.11494	1.75740	11.62969
39	1.64933	1.67025	2.04922	2.59095	2.35238	2.14983	12.46196
41	1.16732	1.98408	2.45163	2.30103	2.58906	2.01368	12.50680
Total	10.02273		13.59843			12.97729	36.59345
Mean	1.67046		2.26640			2.16288	2.03302
Geometric Mean, Mg/cm ²	46.8		184.7			145.5	
M.S. (b)	0.07500		0.04890			0.08134	
S.D. (c)	0.27386		0.22113			0.28520	
C.V., % (d)	8.9		66.4			92.8	

- (a) 1500 °F gas temperature, 0.40 wt. per cent fuel sulfur, 10 ppm "sea salt" in combustor air.
- (b) Mean square (Variance).
- (c) Standard deviation
- (d) Coefficient of variation; from antilogarithm of standard deviation.

From Bartlett's test for homogeneity of variance it is concluded that differences in variance for the three stages are chance variations. Since variance is uniform, statistical analysis of the data should be valid. An analysis of variance to determine the effect of stages and runs on metal loss is shown in Table XXIV.

APPENDIX II (Continued)
TABLE XXIVANALYSIS OF VARIANCE OF LOGARITHMS OF WEIGHT LOSS (MG/CM²) FOR HASTELLOY R-235^(a)

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>	<u>F</u>
Total	17	2.24286		
Runs	2	0.08133	0.04067	0.560
Stages	2	1.21672	0.60836	8.371 ^(b)
Residual (Error)	13	0.94481	0.07268	
Standard Deviation		0.26959		
Coefficient of Variation, %		86.4		

(a) 1500 F gas temperature, 0.40 wt. per cent fuel sulfur, 10 ppm "sea salt" in combustor air.

(b) Differences in metal loss among stages significant (99+ % Confidence).

From this analysis it is concluded that there is no significant difference in the amount of metal loss among runs; however, metal loss varies depending upon the stage of the cascade in which the specimen is tested.

In evaluating the effect of "sea salt" ingestion rate one test was conducted with specimens of Hastelloy R-235 in each position of the cascade at 1500 F gas temperature with 0.40 weight per cent fuel sulfur and 2.0 ppm "sea salt" in combustor air. These data and an analysis of variance are shown in Table XXV.

APPENDIX II (Continued)TABLE XXVREPEATABILITY OF METAL LOSS FOR HASTELLOY R-235 (a)

			Weight Loss		
			Mg/cm ²	Log mg/cm ²	
	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2
	0.7	0.3	0.8	-0.15490	-0.52288
	0.8	0.9	0.8	<u>-0.09691</u>	<u>-0.04576</u>
TOTAL				-0.25181	-0.56864
					-0.19382

Analysis of Variance of Log mg/cm²

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>	<u>F</u>
Total	5	0.15621		
Stages	2	0.04071	0.02035	0.529
Error	3	0.11550	0.03850	

Standard Deviation 0.19622

Coefficient of Variation, % 57.1

(a) 1500 F gas temperature, 0.40 wt. per cent fuel sulfur, 2.0 ppm "sea salt" in combustor air.

From these data it is concluded that the position in the cascade did not effect metal loss. From visual observation of the test specimen the amount of deposit on the specimen was less than for tests with 10.0 ppm "sea salt" which may have reduced the effect of location of the test specimen on metal loss.

One test was conducted with Udimet 700 test specimens in each position in the cascade at 2000 F gas temperature with 0.0002 wt. per cent fuel sulfur and 10.0 ppm "sea salt" in combustor air. These data and an analysis of variance are shown in Table XXVI.

APPENDIX II (Continued)

TABLE XXVI

REPEATABILITY OF METAL LOSS FOR UDIMET-700^(a)

Weight Loss					
Mg/cm ²			Log mg/cm ²		
<u>Stage 1</u>	<u>Stage 2</u>	<u>Stage 3</u>	<u>Stage 1</u>	<u>Stage 2</u>	<u>Stage 3</u>
110.3	92.8	97.5	2.04258	1.96755	1.98900
115.4	129.3	80.4	<u>2.06221</u>	<u>2.11160</u>	<u>1.90526</u>
TOTAL			<u>4.10479</u>	<u>4.07915</u>	<u>3.89426</u>
Mean				2.01303	

Analysis of Variance of Log mg/cm²

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>	<u>F</u>
Total	5	0.02727		
Stages	2	0.01319	0.00660	1.406
Error	3	0.01407	0.00469	
Standard Deviation		0.06849		

Coefficient of Variation, % 17.1

(a) 2000 F gas temperature, 0.0002 wt. per cent fuel sulfur, 10.0 ppm "sea salt" in combustor air.

From this analysis it is concluded that the location of the test specimen does not effect metal loss. Repeatability as measured by coefficient of variation is very good. A visual examination of these test specimens as well as other test specimens exposed to high temperature (2000 F) showed only light scale deposits and a uniform metal attack. It is believed that poorer repeatability and the effect of position of test specimen in the cascade are associated with the accumulation of "sea salt" deposits at temperatures below the fusion point of sodium sulfate (1623 F). Furthermore, poor repeatability was not associated with level of metal loss, as evidenced by the comparable levels of metal loss and wide differences in repeatability for Hastelloy R-235 at 1500 F and Udimet 700 at 2000 F.

APPENDIX II (Continued)

2. Mass Effect of Fuel Sulfur and "Sea Salt".

An examination of the data from the exploratory programs suggests that metal loss may be a mass exposure effect of sulfur and "sea salt". To investigate the possible relationship between metal loss and mass of fuel sulfur and "sea salt" ingested during a test the data reported previously (Ref. 7) with Inconel 713C and Sierra Metal 200 have been reevaluated using multiple regression analysis technique (Ref. 11). Using this technique the relationship between metal loss per unit area and mass of fuel sulfur, "sea salt" and the ratio of "sea salt" to fuel sulfur for a five hour test have been studied. Logarithms of the metal loss and of sulfur, "sea salt" and "sea salt" to sulfur are used in the analyses. Since the addition of a constant to each term will not effect results, unity is added to the mass of fuel sulfur, mass of "sea salt" and the "sea salt" sulfur ratio before converting to logarithms for ease of computation. The geometric means of the metal loss of the four test specimens at each operating condition have been used. Metal loss and mass of reactants for each of the nine operating conditions are shown in Table XXVII. Also shown are the calculated multiple regression equation for the data and calculated metal loss values for each operating condition. A summary of an analysis of variance evaluating the significance of the equation is shown in Table XXVIII. From this analysis it can be concluded at the 99 per cent confidence level that variations in metal loss can be explained on the basis of mass of fuel sulfur, "sea salt" and the ratio of "sea salt" to sulfur.

APPENDIX II (Continued)

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REVIEWS OF PAPERS ON MAIL 273

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$$y = 0.25000 \cdot \frac{1}{x} + 22.00000 \cdot \frac{1}{x^2} - 0.12500 \cdot \frac{1}{x^3} + 0.25000 \cdot \frac{1}{x^4}$$

APPENDIX II (Continued)

TABLE XXVIII

ANALYSIS OF VARIANCE OF METAL LOSS FOR INCONEL 713C

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>	<u>F</u>
Total	8	5.83639966		
Regression	3	5.24319716	1.74773298	14.731 ^(a)
Deviations	5	0.59320250	0.11864050	

(a) The regression is significant at the 99% confidence level.

Data for mass of reactants and weight loss of Sierra Metal 200 from the previous program are shown in Table XXIV. In addition to the nine test points from the previous program which were used to calculate the multiple regression equation, data are included for six tests conducted as part of the exploratory programs. From the analysis of variance which is summarized in Table XXV it can be concluded at the 95 per cent confidence level that the variations in metal loss are associated with changes in the mass of fuel sulfur, "sea salt" and ratio of "sea salt" to sulfur.

TABLE II

TESTS OF INACCURACY AND METAL LOSS FOR SINTERED IRON, 200

Sinter. No. 1	Salt: Mg-Sulfate Mg-Sulfate Mg-Sulfate	Mg-Sulfate Mg-Sulfate Mg-Sulfate	Mg-Sulfate Mg-Sulfate Mg-Sulfate	Y _(a)		Measured Loss, %/cm ²	Calculated Loss, %/cm ²
				Log [$\frac{I_2}{I_1}$: Salt] Log [$\frac{I_2}{I_1}$: Salt] Log [$\frac{I_2}{I_1}$: Salt]	Log [$\frac{I_2}{I_1}$: Salt] Log [$\frac{I_2}{I_1}$: Salt] Log [$\frac{I_2}{I_1}$: Salt]		
0.0002	0	0.00098	0	0.0004254	0	0	0.8314.1
0.0002	1.5	0.00098	0.0011	41.94	0.0174924	1.63286	2.00731
0.0002	15.0	0.00098	0.011	419.4	0.0004254	0.14953	2.37094
0.000	0	0.1%	0	0	0.0777312	0	1.54298
0.000	1.5	0.1%	0.011	0.2097	0.0777312	0.08268	1.43980
0.000	15.0	0.1%	0.411	2.097	0.0777312	0.14953	0.490%
0.40	0	1.9%	0	0	0.47129	0	0
0.40	1.5	1.9%	0.0411	0.03097	0.47129	0.009013	1.16104
0.40	15.0	1.9%	0.411	0.2097	0.47129	0.08268	1.40568
Total				1.6483398	0.5010672	4.921833	13.60851
Mean				0.1851169	0.055674	0.546870	1.51206
						Data from samples 1927, 2000, not used in regression analysis	
0.0002	20.0	0.0009	0.270	300.0	0.0003907	0.10380	2.47857
0.40	20.0	1.60	0.270	0.190	0.4716	0.10380	0.06070
0.0002	20.0	0.000208	0.07425	317.1	0.000469	0.0116255	2.50256
0.0002	20.0	0.000312	0.070	321.2	0.001390	0.0374.3	2.15685
0.0002	20.0	0.000308	0.270	290.0	0.000468	0.10380	2.40157
0.0002	20.0	0.00098	0.135	250.0	0.0002245	0.0549959	2.39987

(a) Calculated Polynomials

Calculated multiple regression equation is: $\hat{Y} = -0.83699 X_1 + 3.50688 X_2 + 0.25689 X_3 + 1.33001$

APPENDIX II (Continued)

TABLE XX

ANALYSIS OF VARIANCE OF METAL LOSS FOR SIERRA METAL 200

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>	<u>F</u>
Total	8	2.49710598		
Regression	3	2.00385402	0.66795134	6.771 (a)
Deviation	5	0.49325196	0.09865039	

(a) The regression is significant at the 95% confidence level.

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